NASA Conference Publication 3304

National Educators' Workshop: Update 94

Standard Experiments in Engineering Materials Science and Technology

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PREFACE

NEW:Update 94 was held November 7 - 9, 1994, at National Institute of Standards and Technology (NIST) in Gaithersburg, Maryland. Building on past workshops and extensive workshop evaluations, this **9th annual NEW:Update** continued to follow the theme of strengthening materials education. Over 130 participants witnessed demonstrations of experiments, discussed issues of materials science and engineering (MS&E) with people from education, industry, government, and technical societies, heard about new MS&E developments, and attended 3 hour mini workshops in state-of-the-art NIST laboratories. Concurrent sessions were held in order to accommodate all of the demonstrations of experiments. Faculty in attendance represented high schools, community colleges, smaller colleges, and major universities. Undergraduate and graduate students also attended.

Representing the American Society for Engineering Education's (ASEE) Materials Division, Dave Werstler presented a special plaque to NIST personnel Joniee Adams, Anna Fraker, Brian Belanger, Harry Rook, and Lyle Schwartz for their valuable contributions to MS&E education through their nine years of support to **NEW:Updates** and other materials education efforts. The overwhelming success of **NEW:Updates** results in a large measure from the considerable work and sacrifices of the host agencies: NIST, NASA Langley Research Center, Oak Ridge National Laboratory, and Norfolk State University.

NEW:Update 94 participants aided in evaluating over thirty-five experiments that were presented before the group. Additional updating information relating to materials science, engineering and technology was also presented as mini plenary sessions that focused on technology transfer from NIST, design with Continuous Fiber Ceramic Composites (CFCCs),intelligent processing, nanostructured materials, emerging materials technology and ASM International's educational tools. We also learned about the technology and advantages of CD ROM for strengthening materials education and of joint effort among ASEE, Materials Education Council (MEC) and NSU to develop CD ROM disks of the **NEW:Updates**. You will find transparency masters for the mini plenary sessions included in this publication.

The experiments in this publication can serve as a valuable guide to faculty who are interested in useful activities for their students. The material was the result of years of research aimed at better methods of teaching materials science, engineering and technology. The experiments were developed by faculty, scientists, and engineers throughout the United States. There is a blend of experiments on new materials and traditional materials. Uses of computers in MS&E, experimental design, and a variety of low cost experiments were among the demonstrations presented. Transparency masters of technical presentations are also included.

Experiments underwent an extensive peer review process. After submission of abstracts, selected authors were notified of their acceptance and given the format for submission of experiments. Experiments were reviewed by a panel of specialists through the cooperation of the Materials Education Council. Authors received comments from the panel prior to NEW:Update 94, allowing them to make necessary adjustments prior to demonstrating their experiments. Comments from workshop participants provided additional feedback which authors used to make final revisions which were submitted for the NASA editorial group for this publication.

The Materials Education Council of the United States publishes selected experiments in the ***Journal of Materials Education*** (***JOE***). The ***JOE*** offers materials educators valuable teaching and curriculum aids including instructional modules on emerging materials technology, experiments, book reviews, and editorials.

Videotapes were made of the workshop by NIST with assistance from Robert Berrettini and Alfred E. McKenney. As with previous NEW:Updates, critiques were made of the workshop to provide continuing improvement of this activity. The evaluations and recommendations made by participants provide valuable feedback for planning of subsequent **NEW:Updates.**

NEW:Update 94 and the series of workshops that go back to 1986 are, to our knowledge, the only national workshops or gatherings for materials educators that have a focus on the full range of issues on strategies for better teaching about the full complement of materials. Recognizing the problem of motivating young people to pursue careers in MSE, we have included exemplary pre-university activities such as Adventures in Science, ASM International Education Foundation's Career Outreach Program, Engineers for Education, National Teachers Institute for Materials Science and Technology, and several programs run through high schools.

NEW:Update 94, with its diversity of faculty, industry, and government MSE participants, served as a forum for both formal and informal issues facing MSE education that ranged from the challenges of keeping faculty and students abreast of new technology to ideas to insure that materials scientists, engineers, and technicians maintain the proper respect for the environment in the pursuit of their objectives.

NEW:Update 94 resulted from considerable cooperative efforts by individuals in government, education, and industry. The workshop's goal is to maintain the network of participants and to continue to collect these ideas and resources to bring them together in a compendium of experiments in materials science, engineering and technology. Please read the paper in this book , "CD ROM Technology to Strengthen Materials Education", to learn more about efforts to develop CD ROM compendium disks.

We hope that the experiments presented in this publication will assist you in teaching about materials science, engineering and technology. We would like to have your comments on their value and means of improving them. Please send comments to James A. Jacobs, School of Technology, Norfolk State University, Norfolk, Virginia 23504.

We express our appreciation to all those who helped to keep this series of workshops viable.

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CONTENTS

REVIEWERS FOR NEW:Update 94

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Technical notebooks and announcements of the workshop were provided by
NASA LANGLEY RESEARCH CENTER

NIST MINI WORKSHOPS

♦ **Intelligent Processing of Materials - Building 231, Room 8154**
Stephen D. Ridder, Frank Biancaniello, Michael Zachariah, and Rodney Jiggetts

State of the art computer systems are used in metallurgical processes such as gas atomization, and image analysis. Control, feedback and analysis will be presented.

♦ **Automated Manufacturing Research Facility - Building 304 AMRF/Shop Floor**
Denver Lovett

Shops of Future Manufacturing - This workshop will explore the role of information technology and multimedia in developing high performance training systems for manufacturing.

♦ **Ceramic Materials by Design - Building 223, Room A-258**
John Blendell and Carol Handwerker

Use of high technology ceramic materials requires carefully controlled processing in order to exploit their unique properties. The relationship between ceramic microstructure and properties will be described as well as processing routes to control microstructure.

♦ **Growth, Fabrication and Testing of Novel Semiconductor Devices - Building 221, Room A 151**
Curt Richter

It will be shown how semiconductors are grown by molecular beam epitaxy techniques and then are fabricated into specialized devices and tested.

♦ **Magnetic Nanocomposites - Building 223, Room B 143**
Joseph Ritter and Robert D. Shull

Preparation and property measurement of composites with constituents only a few nanometers will be demonstrated.

♦ **Polymer Composites - Building B 224, Room A 210**
Donald Hunston

Methods of reducing fabrication costs and basic studies of degradation mechanisms for life prediction will be presented.

♦ **Optical Tweezers - Building 221, Room A-164**
Kristian Helmerson

Tightly focused beams of laser light can be used to remotely manipulate microscopic objects, including biological material. Such optical traps, commonly referred to as "optical tweezers" hold great promise for noninvasive micromanipulation and mechanical measurement in biology.

♦ **X-ray Optics and Multilayer Microstructures: Design, Production, Testing and Applications - Building 221, Room A-246 and A-47**
Joseph Pedulla, Richard Watts, Thomas Lucatorto, and Richard D. Deslattes

Provides a view of a new technology: the fabrication of soft x-ray mirrors by deposition of multilayer microstructures. Normally, soft x-rays are absorbed when incident upon material surfaces. By careful design and deposition of alternate layers of high density and low density materials, one can exploit subtle interference effects to achieve remarkable reflectivities throughout the range of 9-20 nm wavelength. Discussion of how these mirrors are constructed, evaluated, and applied in various situations.

♦ **Plasma Processing of Materials - Building 221, Room A-366**
James R. Roberts
Utilizing the GEC rf reference cell as an experimental platform, investigations of plasma conditions in this reproducible source are brought to bear on process control. Correlation of optical emission spectroscopy and laser induced fluorescencet may provide a simple plasma reproducibility and stability monitor for semiconductor etching.

♦ **Surface Analysis - Building 222, Room B-133**
Dale Newbury
Microscopes based on beams of electrons, ions, or photons provide high resolution morphological visualization (to nanometer scales) combined with elemental and molecular analysis. These techniques are used to characterize the microstructure of matter and to elucidate processes fundamental to the physical and biological sciences as well as to technology.

♦ **Light Scattering from Optical Materials - Building 220 (Metrology) Room A-320**
Clara Asmail
The way in which a surface scatters incident light reveals information about the microroughness of the surfact topography. The bidirectional reflectance distribution function (BRDF). Industrial applications of BRDF will be explored.

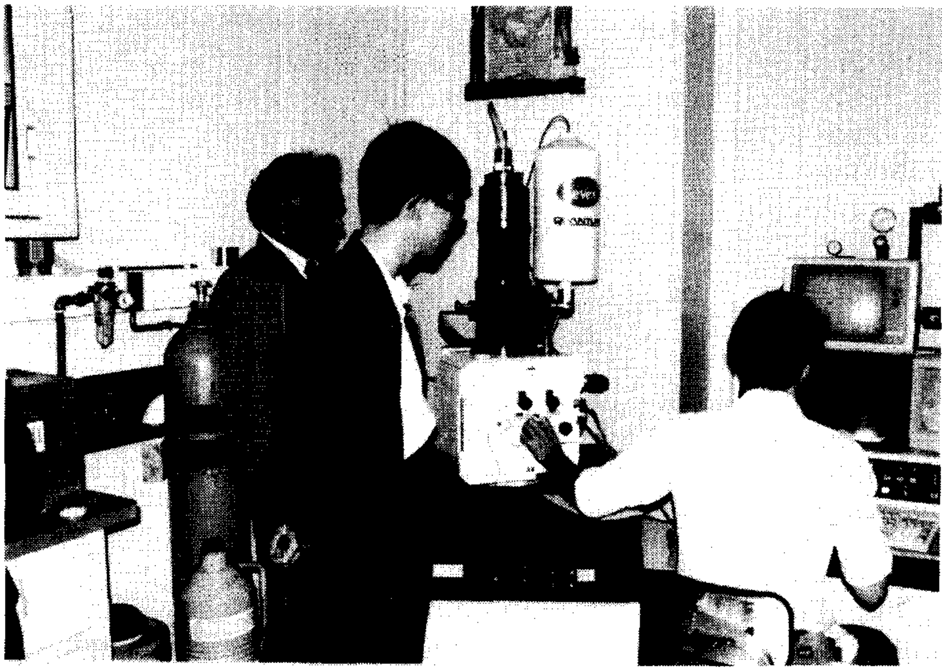
♦ **Mechanical Testing - Building 202, High Bay Area**
Richard Field
A review of mechanical testing and mechanical properties of materials will be given. Detailed discussion will be given of testing done at the NIST on small specimens that are tested in the transmission electron microscope and on larger specimens that are tested in the 12 million pound (53MN) test facility.

♦ **Industrial Radiation Processing - Building 245, Room C-209**
Marc F. Desrosiers, William L. McLaughlin, and Marlon L. Walker
An overview with demonstration will be given on ionizing radiation and how it is used in industry, including foods, medical devices, drugs, wastes, etc. and the role of NIST's measurement services.

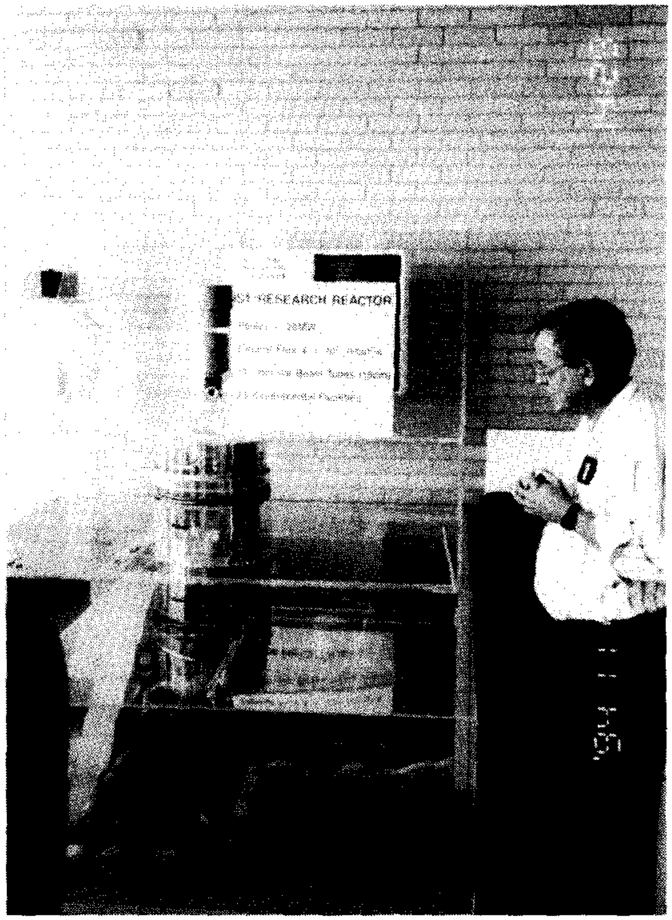
♦ **National Facility Tour: Cold Neutron Research Facility - Building 235, Lobby**
Henry Prask
Instrumentation, representative measurements and information on how qualified members of the scientific community could use the facility will be presented.

♦ **Development and evaluation of Dental Adhesive Materials - Building 224, Room A-149**
Laurie George
The development of dental adhesives used to bond dental restorative materials to teeth involves the application of organic chemistry, analytical testing, physical testing, and biological testing.

♦ **SQUID - Building 101, Lecture Room D**
Richard Kerchner
Construction of an easily produced Superconducting Quantum Interference Device and demonstrations both of its sensitivity to magnetic fields and its usefulness as an electronic logic component.



Mini Workshops



Mini Workshops

LISTING OF EXPERIMENTS FROM NEW:UPDATES

EXPERIMENTS & DEMONSTRATIONS IN STRUCTURES, TESTING, AND EVALUATION

NEW:Update 88 **NASA Conference Publication 3060**

Sastri, Sankar. "Fluorescent Penetrant Inspection"
Sastri, Sankar. "Magnetic Particle Inspection"
Sastri, Sankar. "Radiographic Inspection"

NEW:Update 89 **NASA Conference Publication 3074**

Chowdhury, Mostafiz R. and Chowdhury, Farida. "Experimental Determination of Material Damping Using Vibration Analyzer"
Chung, Wenchiang R. "The Assessment of Metal Fiber Reinforced Polymeric Composites"
Stibolt, Kenneth A. "Tensile and Shear Strength of Adhesives"

NEW:Update 90 **NIST Special Publication 822**

Azzara, Drew C. "ASTM: The Development and Application of Standards"
Bates, Seth P. "Charpy V-Notch Impact Testing of Hot Rolled 1020 Steel to Explore Temperature Impact Strength Relationships"
Chowdhury, Mostafiz R. "A Nondestructive Testing Method to Detect Defects in Structural Members"
Cornwell, L. R., Griffin, R. B., and Massarweh, W. A. "Effect of Strain Rate on Tensile Properties of Plastics"
Gray, Stephanie L., Kern, Kristen T., Harries, Wynford L., and Long, Sheila Ann T. "Improved Technique for Measuring Coefficients of Thermal Extension for Polymer Films"
Halperin, Kopl. "Design Project for the Materials Course: To Pick the Best Material for a Cooking Pot"
Kundu, Nikhil. "Environmental Stress Cracking of Recycled Thermoplastics"
Panchula, Larry and Patterson, John W. "Demonstration of a Simple Screening Strategy for Multifactor Experiments in Engineering"
Taylor, Jenifer A. T. "How Does Change in Temperature Affect Resistance?"
Wickman, Jerry L. and Corbin, Scott M. "Determining the Impact of Adjusting Temperature Profiles on Photodegradability of LDPE/Starch Blown Film"
Widener, Edward L. "It's Hard to Test Hardness"
Widener, Edward L. "Unconventional Impact-Toughness Experiments"

NEW:Update 91 **NASA Conference Publication 3151**

Bunnell, L. Roy. "Tempered Glass and Thermal Shock of Ceramic Materials"
Lundeen, Calvin D. "Impact Testing of Welded Samples"
Gorman, Thomas M. "Designing, Engineering, and Testing Wood Structures"
Strehlow, Richard R. "ASTM - Terminology for Experiments and Testing"
Karplus, Alan K. "Determining Significant Material Properties, A Discovery Approach"
Spiegel, F. Xavier and Weigman, Bernard J. "An Automated System for Creep Testing"
Denton, Nancy L. and Hillsman, Vernon S. "Isotropic Thin-Walled Pressure Vessel Experiment"
Allen, David J. "Stress-Strain Characteristics of Rubber-Like Materials: Experiment and Analysis"
Dahl, Charles C. "Computer Integrated Lab Testing"
Cornwell, L. R. "Mechanical Properties of Brittle Material"

NEW:Update 92

Bunnell, L. Roy. "Temperature-Dependent Electrical Conductivity of Soda-Lime Glass and Construction and Testing of Simple Airfoils to Demonstrate Structural Design, Materials Choice, and Composite Concepts"

Marpet, Mark I. "Walkway Friction: Experiment and Analysis"

Martin, Donald H. "Application of Hardness Testing in Foundry Processing Operations: A University and Industry Partnership"

Masi, James V. "Experiments in Corrosion for Younger Students By and For Older Students"

Needham, David. "Micropipet Manipulation of Lipid Membranes: Direct Measurement of the Material Properties of a Cohesive Structure That is Only Two Molecules Thick"

Perkins, Steven W. "Direct Tension Experiments on Compacted Granular Materials"

Shih, Hui-Ru. "Development of an Experimental Method to Determine the Axial Rigidity of a Strut-Node Joint"

Spiegel, F. Xavier. "An Automated Data Collection System For a Charpy Impact Tester"

Tipton, Steven M. "A Miniature Fatigue Test Machine"

Widener, Edward L. "Tool Grinding and Spark Testing"

NEW:Update 93 **NASA Conference Publication 3259**

Borst, Mark A. "Design and Construction of a Tensile Tester for the Testing of Simple Composites"

Clum, James A. "Developing Modules on Experimental Design and Process Characterization for Manufacturing/Materials Processes Laboratories"

Diller, T. E. and A. L. Wicks, "Measurement of Surface Heat Flux and Temperature"

Denton, Nancy and Vernon S. Hillsman, "An Introduction to Strength of Materials for Middle School and Beyond"

Fisher, Jonathan H. "Bridgman Solidification and Experiment to Assess Boundaries and Interface Shape"

Gray, Jennifer "Symmetry and Structure Through Optical Diffraction"

Karplus, Alan K. "Knotty Knots"

Kohne, Glenn S. "An Automated Digital Data Collection and Analysis System for the Charpy Impact Tester"

Olesak, Patricia J. "Scleroscope Hardness Testing"

Speigel, F. Xavier, "Innexpensive Materials Science Demonstrations"

Wickman, J. L. "Plastic Part Design Analysis Using Polarized Filters and Birefringence"

Widener, Edward L. "Testing Rigidity by Torque Wrench"

EXPERIMENTS & DEMONSTRATIONS IN METALS

NEW:Update 88 **NASA Conference Publication 3060**

Nagy, James P. "Sensitization of Stainless Steel"

Neville, J. P. "Crystal Growing"

Pond, Robert B. "A Demonstration of Chill Block Melt Spinning of Metal"

Shull, Robert D. "Low Carbon Steel: Metallurgical Structure vs. Mechanical Properties"

NEW:Update 89 **NASA Conference Publication 3074**

Balsamel, Richard. "The Magnetization Process - Hysteresis"

Beardmore, Peter. "Future Automotive Materials - Evolution or Revolution"

Bunnell, L. Roy. "Hands-On Thermal Conductivity and Work-Hardening and Annealing in Metals"

Kazem, Sayyed M. "Thermal Conductivity of Metals"

Nagy, James P. "Austempering"

NEW:Update 90 **NIST Special Publication 822**

Bates, Seth P. "Charpy V-Notch Impact Testing of Hot Rolled 1020 Steel to Explore Temperature Impact Strength Relationships"
Chung, Wenchiang R. and Morse, Margery L. "Effect of Heat Treatment on a Metal Alloy"
Rastani, Mansur. "Post Heat Treatment in Liquid Phase Sintered Tungsten-Nickel-Iron Alloys"
Spiegel, F. Xavier. "Crystal Models for the Beginning Student"
Yang, Y. Y. and Stang, R. G. "Measurement of Strain Rate Sensitivity in Metals"

NEW:Update 91 **NASA Conference Publication 3151**

Cowan, Richard L. "Be-Cu Precipitation Hardening Experiment"
Kazem, Sayyed M. "Elementary Metallography"
Krepski, Richard P. "Experiments with the Low Melting Indium-Bismuth Alloy System"
Lundeen, Calvin D. "Impact Testing of Welded Samples"
McCoy, Robert A. "Cu-Zn Binary Phase Diagram and Diffusion Couples"
Patterson, John W. "Demonstration of Magnetic Domain Boundary Movement Using an Easily Assembled Videocam-Microscope System"
Widener, Edward L. "Heat-Treating of Materials"

NEW:Update 92

Dahiya, Jai N. "Phase Transition Studies in Barium and Strontium Titanates at Microwave Frequencies"
Rastani, Mansur. "Improved Measurement of Thermal Effects on Microstructure"
Walsh, Daniel W. "Visualizing Weld Metal Solidification Using Organic Analogs"

NEW:Update 93 **NASA Conference Publication 3259**

Guichelaar, Philip J. "The Anisotrophy of Toughness in Hot-Rolled Mild Steel"
Martin, Donald H. "From Sand Casting TO Finished Product (A Basic University-Industry Partnership)"
Petit, Jocelyn I. "New Developments in Aluminum for Aircraft and Automobiles"
Smith, R. Carlisle "Crater Cracking in Aluminum Welds"

EXPERIMENTS & DEMONSTRATIONS IN POLYMERS

NEW:Update 89 **NASA Conference Publication 3074**

Chung, Wenchiang R. "The Assessment of Metal Fiber Reinforced Polymeric Composites"
Greet, Richard and Cobaugh, Robert. "Rubberlike Elasticity Experiment"
Kern, Kristen T., Harries, Wynford L., and Long, Sheila Ann T. "Dynamic Mechanical Analysis of Polymeric Materials"
Kundu, Nikhil K. and Kundu, Malay. "Piezoelectric and Pyroelectric Effects of a Crystalline Polymer"
Kundu, Nikhil K. "The Effect of Thermal Damage on the Mechanical Properties of Polymer Regrinds"
Stibolt, Kenneth A. "Tensile and Shear Strength of Adhesives"
Widener, Edward L. "Industrial Plastics Waste: Identification and Segregation"
Widener, Edward L. "Recycling Waste-Paper"

NEW:Update 90 **NIST Special Publication 822**

Brostow, Witold and Kozak, Michael R. "Instruction in Processing as a Part of a Course in Polymer Science and Engineering"

Cornwell, L. R., Griffin, R. B., and Massarweh, W. A. "Effect of Strain Rate on Tensile Properties of Plastics"

Gray, Stephanie L., Kern, Kristen T., Harries, Wynford L., and Long, Sheila Ann T. "Improved Technique for Measuring Coefficients of Thermal Extension for Polymer Films"

Humble, Jeffrey S. "Biodegradable Plastics: An Informative Laboratory Approach"

Kundu, Nikhil. "Environmental Stress Cracking of Recycled Thermoplastics"

Wickman, Jerry L. and Corbin, Scott M. "Determining the Impact of Adjusting Temperature Profiles on Photodegradability of LDPE/Starch Blown Film"

NEW:Update 91 **NASA Conference Publication 3151**

Allen, David J. "Stress-Strain Characteristics of Rubber-Like Materials: Experiment and Analysis"

Chowdhury, Mostafiz R. "An Experiment on the Use of Disposable Plastics as a Reinforcement in Concrete Beams"

Gorman, Thomas M. "Designing, Engineering, and Testing Wood Structures"

Lloyd, Isabel K., Kolos, Kimberly R., Menegaux, Edmond C., Luo, Huy, McCuen, Richard H., and Regan, Thomas M. "Structure, Processing and Properties of Potatoes"

McClelland, H. T. "Laboratory Experiments from the Toy Store"

Sorensen, Carl D. "Measuring the Surface Tension of Soap Bubbles"

Wickman, Jerry L. and Plocinski, David. "A Senior Manufacturing Laboratory for Determining Injection Molding Process Capability"

NEW:Update 92

Kundu, Nikhil K. "Performance of Thermal Adhesives in Forced Convection"

Liu, Ping. "Solving Product Safety Problem on Recycled High Density Polyethylene Container"

Wickman, Jerry L. "Thermoforming From a Systems Viewpoint"

NEW:Update 93 **NASA Conference Publication 3259**

Csernica, Jeffrey "Mechanical Properties of Crosslinked Polymer Coatings"

Edblom, Elizabeth "Testing Adhesive Strength" & "Adhesives The State of the Industry"

Elban, Wayne L. "Three-Point Bend Testing of Poly (Methyl Methacrylate) and Balsa Wood"

Labana, S. S. "Recycling of Automobiles an Overview"

Liu, Ping and Tommy L. Waskom, "Application of Materials Database (MAT.DB>) to Materials Education and Laminated Thermoplastic Composite Material"

Marshall, John A. "Liquids That Take Only Milliseconds to Turn into Solids"

Quaal, Karen S. "Incorporating Polymeric Materials Topics into the Undergraduate Chemistry Cor Curriculum: NSF-Polyed Scholars Project: Microscale Synthesis and Characterization of Polystyrene"

EXPERIMENTS & DEMONSTRATIONS IN CERAMICS

NEW:Update 88 **NASA Conference Publication 3060**

Nelson, James A. "Glasses and Ceramics: Making and Testing Superconductors"

Schull, Robert D. "High T_c Superconductors: Are They Magnetic?"

NEW:Update 89 **NASA Conference Publication 3074**
Beardmore, Peter. "Future Automotive Materials - Evolution or Revolution"
Bunnell, L. Roy. "Hands-On Thermal Conductivity and Work-Hardening and Annealing in Metals"
Link, Bruce. "Ceramic Fibers"
Nagy, James P. "Austempering"
Ries, Heidi R. "Dielectric Determination of the Glass Transition Temperature"

NEW:Update 90 **NIST Special Publication 822**
Dahiya, J. N. "Dielectric Behavior of Superconductors at Microwave Frequencies"
Jordan, Gail W. "Adapting Archimedes' Method for Determining Densities and Porosities of Small Ceramic Samples"
Snail, Keith A., Hanssen, Leonard M., Oakes, David B., and Butler, James E. "Diamond Synthesis with a Commercial Oxygen-Acetylene Torch"

NEW:Update 91 **NASA Conference Publication 3151**
Bunnell, L. Roy. "Tempered Glass and Thermal Shock of Ceramic Materials"
Craig, Douglas F. "Structural Ceramics"
Dahiya, J. N. "Dielectric Behavior of Semiconductors at Microwave Frequencies"
Weiser, Martin W., Lauben, David N., and Madrid, Philip. "Ceramic Processing: Experimental Design and Optimization"

NEW:Update 92
Bunnell, L. Roy. "Temperature-Dependent Electrical Conductivity of Soda-Lime Glass"
Henshaw, John M. "Fracture of Glass"
Stephan, Patrick M. "High Thermal Conductivity of Diamond"
Vanasupa, Linda S. "A $.69 Look at Thermoplastic Softening"

NEW:Update 93 **NASA Conference Publication 3259**
Bunnell, L. Roy and Stephen Piippo, "Property Changes During Firing of a Typical Porcelain Ceramic"
Burchell, Timothy D. "Developments in Carbon Materials"
Dahiya, J.N., "Dieletric Measurements of Selected Ceramics at Microwave Frequencies"
Ketron, L.A. "Preparation of Simple Plaster Mold for SLip Casting and Slip Casting"
Masi, James V. "Experiments in Diamond Film Fabrication in Table Top Plasma Apparatus"
Werstler, David E. "Microwave Sintering of Machining Inserts"

EXPERIMENTS & DEMONSTRATIONS IN COMPOSITES

NEW:Update 88 **NASA Conference Publication 3060**
Nelson, James A. "Composites: Fiberglass Hand Laminating Process"

NEW:Update 89 **NASA Conference Publication 3074**
Beardmore, Peter. "Future Automotive Materials - Evolution or Revolution"
Chung, Wenchiang R. "The Assessment of Metal Fiber Reinforced Polymeric Composites"
Coleman, J. Mario. "Using Template/Hotwire Cutting to Demonstrate Moldless Composite Fabrication"

NEW:Update 90 **NIST Special Publication 822**
Bunnell, L. R. "Simple Stressed-Skin Composites Using Paper Reinforcement"
Schmenk, Myron J. "Fabrication and Evaluation of a Simple Composite Structural Beam"
West, Harvey A. and Sprecher, A. F. "Fiber Reinforced Composite Materials"

NEW:Update 91 **NASA Conference Publication 3151**
Greet, Richard J. "Composite Column of Common Materials"

NEW:Update 92
Thornton, H. Richard. "Mechanical Properties of Composite Materials"

NEW:Update 93 **NASA Conference Publication 3259**
Masters, John "ASTM Methods for Composite Characterization and Evaluation"
Webber, M. D. and Harvey A. West. "Continuous Unidirectional Fiber Reinforced Composites: Fabrication and Testing"

EXPERIMENTS & DEMONSTRATIONS IN ELECTRONIC MATERIALS

NEW:Update 88 **NASA Conference Publication 3060**
Sastri, Sankar. "Magnetic Particle Inspection"

NEW:Update 89 **NASA Conference Publication 3074**
Kundu, Nikhil K. and Kundu, Malay. "Piezoelectric and Pyroelectric Effects of a Crystalline Polymer"
Molton, Peter M. and Clarke, Clayton. "Anode Materials for Electrochemical Waste Destruction"
Ries, Heidi R. "Dielectric Determination of the Glass Transition Temperature"

NEW:Update 90 **NIST Special Publication 822**
Dahiya, J. N. "Dielectric Behavior of Superconductors at Microwave Frequencies"

NEW:Update 91 **NASA Conference Publication 3151**
Dahiya, J. N. "Dielectric Behavior of Semiconductors at Microwave Frequencies"
Patterson, John W. "Demonstration of Magnetic Domain Boundary Movement Using an Easily Assembled Videocam-Microscope System"

NEW:Update 92
Bunnell, L. Roy. "Temperature-Dependent Electrical Conductivity of Soda-Lime Glass
Dahiya, Jai N. "Phase Transition Studies in Barium and Strontium Titanates at Microwave Frequencies"

EXPERIMENTS & TOPICS IN MATERIALS CURRICULUM

NEW:Update 93 **NASA Conference Publication 3259**
Bright, Victor M. "Simulation of Materials Processing: Fantasy or Reality?"
Diwan, Ravinder M. "Manufacturing Processes Laboratory Projects in Mechanical Engineering Curriculum"
Kundu, Nikhil K. "Graphing Techniques for Materials Laboratory Using Excel"
McClelland, H. T. "Process Cability Determination of New and Existing Equipment and Introduction to Usable Statistical Methods"
Passek, Thomas "University Outreach Focused Discussion: What Do Educators Want From ASM International"

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or

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Cost = $30.00

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Classic Crystals: A Book of Models - Hands-on Morphology. Twenty-Four Common Crystal models to assemble and study. Aids in learning symmetry and Miller indices. $19.00.

A Set of Four Hardbound Volumes of Wood Modules - The Clark C. Heritage Memorial Series. Published by MEC in cooperation with the U.S.Forest Products Laboratory, Madison, Wisconsin. A compilation of nine modules entitled Wood: Its Structure and Properties (I), edited by Frederick F. Wangaard. A compilation of eight modules especially developed for architects and civil engineers entitled Wood As A Structural Material (II). Also, Adhesive Bonding of Wood and Other Structural Materials III and Wood: Engineering Design Concepts (IV). Each of the first three wood volumes costs $29.00; the fourth volume costs $39.00. The entire four-volume set is only $ 120.00 plus $3.50 shipping ($5.50 overseas).

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Metallographic Atlas - Royal Swedish Institute of Technology. $31.00. A brief introduction to the microstructures of metallic materials - how they appear and how they can be modified.

Please add $2.50 per book shipping charge.
Checks payable to The Pennsylvania State University

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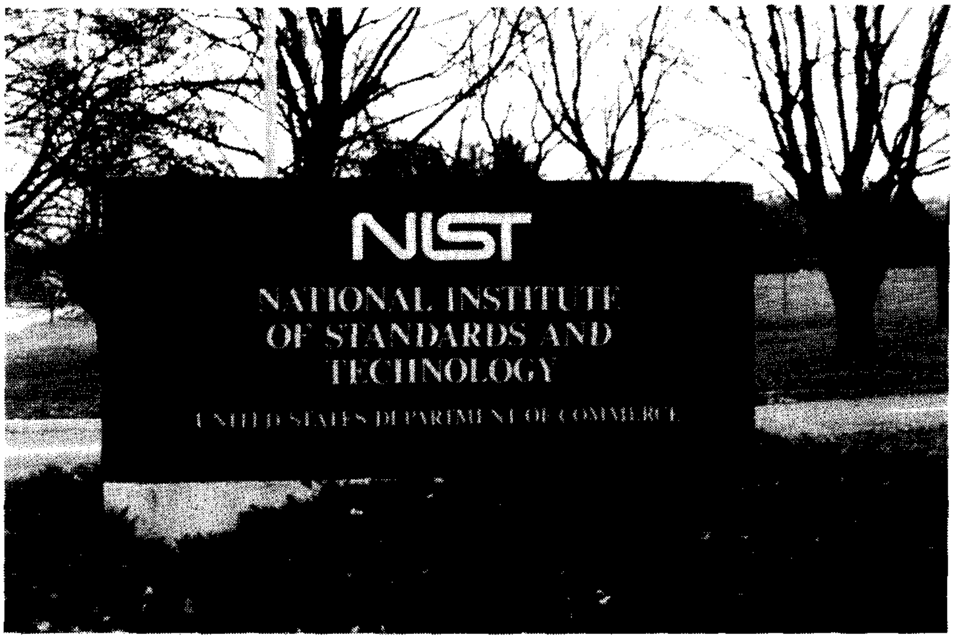

Workshop Location

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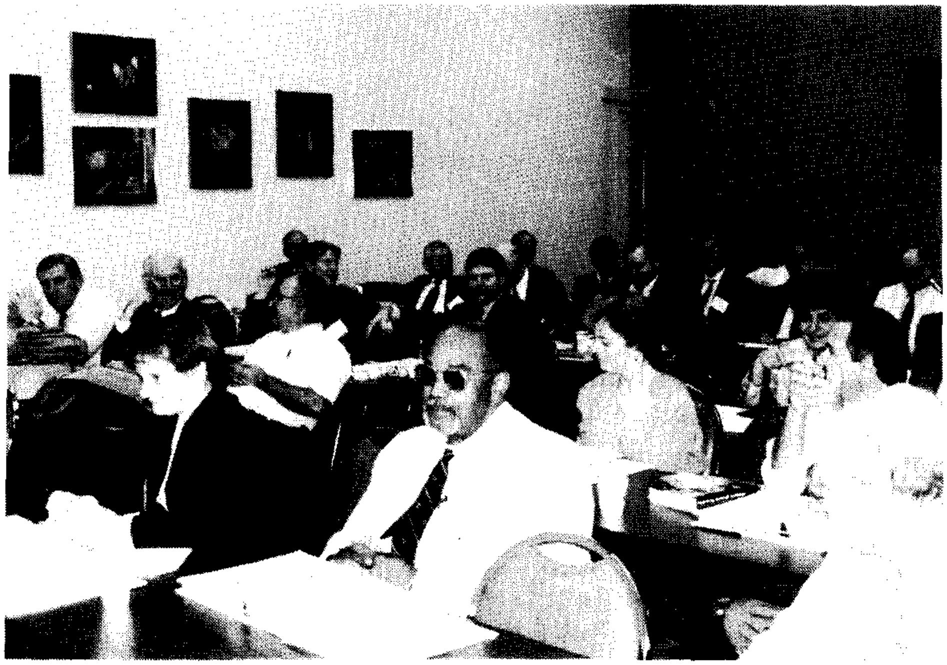

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Recognizing Contributions

Recognizing Contributions

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AGENDA

- Historical Perspectives
- Lessons from Abroad
- Congressional Actions
- Experiences at NIST

HISTORICAL PERSPECTIVES

- World Leadership Through the 1960s
- Declining Market Share in 1970s
- Government Roles in Civilian Technology Developed in 1980s
- Congressional Leadership

LESSONS FROM ABROAD

- Technology Development;
  Industry Led, Government Lab Supported
- Technology Deployment;
  Small Business Focus in Japan and Europe

CONGRESSIONAL ACTIONS

- 1980 - Stevenson-Wydler
- 1984 - Joint R&D Partnerships
- 1986 - GoGo Labs Given CRADA Authority
- 1987 - Malcolm Baldrige Quality Award Initiated
- 1988 - Technololgy Competitiveness Act
  Creates NIST, ATP, MTC
- 1992 - Technology Reinvestment Program
- 1993 - S.4, H.R. 820 Pending

PRESIDENTIAL ACTION

- 1993 - President Clinton targets DoC and NIST for major role in assisting industry, calls for significant budget growth.

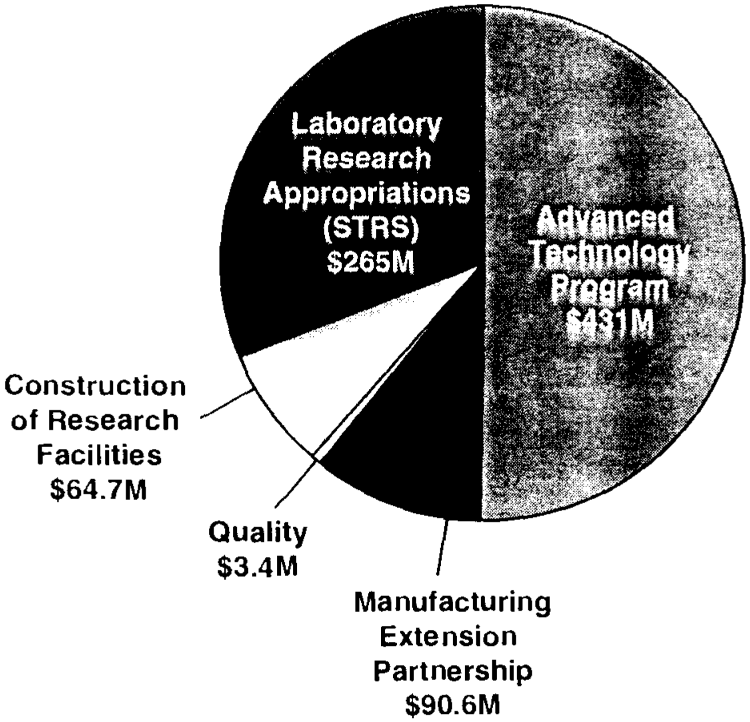
WHAT DOES NIST DO?

- Fundamental Research
- Develop New Measurement Techniques and Devices
- Characterize Materials , Products, and Systems
- Evaluate and Publish Standard Reference Data
- Provide Standard Reference Materials
- Provide Measurement Assurance Services
- Develop and Propose Product Standards and Code Provisions
- Award Grants to U.S. Industry for Pre-Competitive Technology
- Demonstrate the Results
  - Translate and Transfer to Users
  - Manufacturing Technology Centers
- Manage the Malcolm Baldrige National Quality Award

NIST DIRECT APPROPRIATIONS

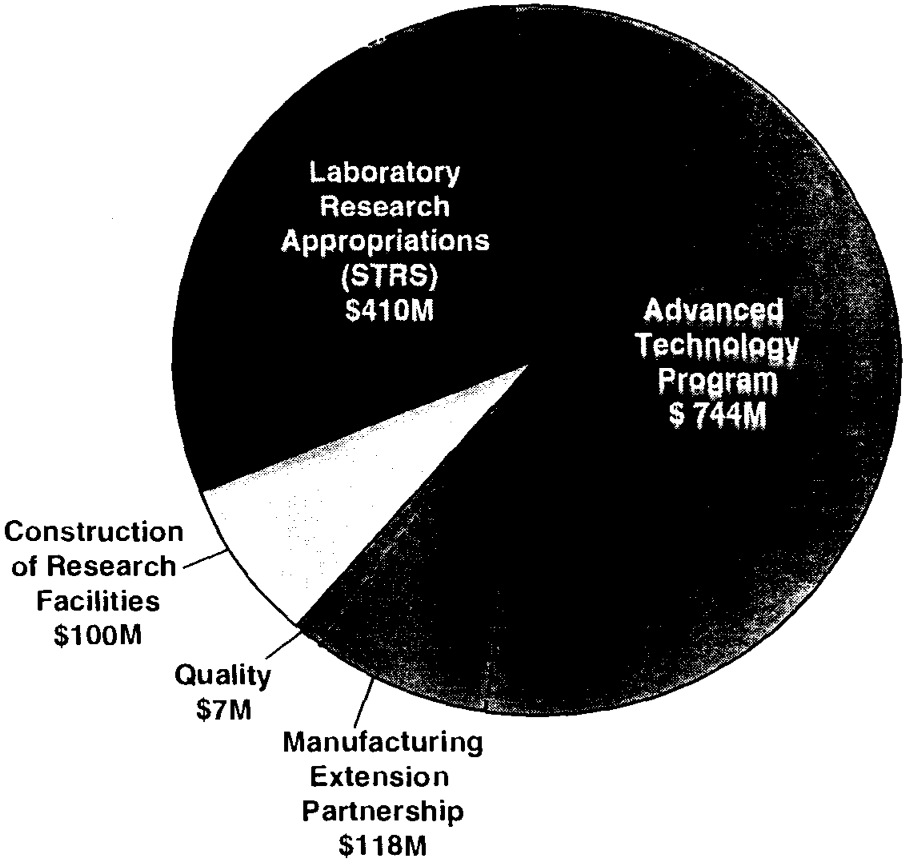
President's Plan

FY 1995 Actual



Total Appropriations = $ 854.7M

FY 1997 Proposal



Total Appropriations = $1,379M

EXPERIENCES AT NIST

- Advanced Technology Program
- Manufacturing Extension Partnership
- NIST Laboratory Program

ATP CHARACTERISTICS

- Unique Mission Focus — High-Risk Enabling Technology Development to Stimulate U.S. Economic Growth
- Partnership with Industry — Industry Conceives and Proposes Ideas, Executes and Cost-Shares Projects; ATP Applies Selection Criteria
- Focused and General Competitions — for Depth and Breadth
- Competitive Selection Process — Technical and Business Reviews
- Sunset Provisions — for Both Programs and Projects
- Performance Metrics — for Both Process and Programs

ADVANCED TECHNOLOGY PROGRAM

Focused Program Areas

First Round: April, 1994

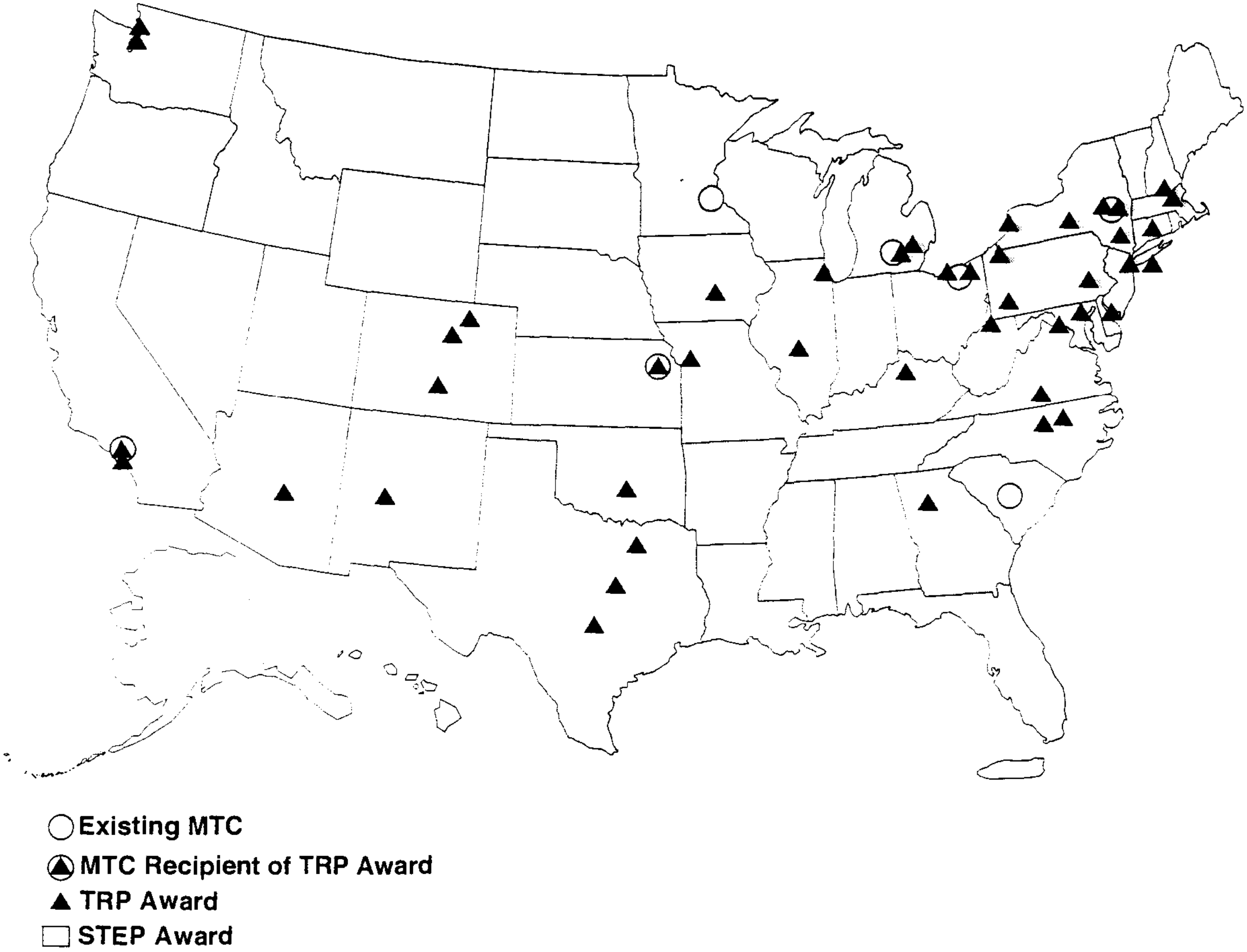
- Manufacturing for Composites
- Computer Integrated Manufacturing for Electronics
- Health Care Information Infrastructure
- DNA Diagnostic Tools
- Component Level Software

MANUFACTURING EXTENSION PARTNERSHIP

NIST is building the partnership on four major elements:

- Manufacturing Technology Centers (MTCs)
- Manufacturing Outreach Centers (MOCs)
- the State Technology Extension Program (STEP)
- LINKS - a support, coordination and linking/ networking infrastructure

NIST MANUFACTURING EXTENSION PARTNERSHIP (Including MTCs, STEP Awards, and TRP Projects Managed by NIST)



NIST AND THE TECHNOLOGY REINVESTMENT PROGRAM

- Participation in Evaluation
- TRP and the MEP

MATERIALS SCIENCE AND ENGINEERING LABORATORY

MSEL

MSEL MISSION

Stimulate the more effective production and use of materials by working with industry in the development of technology, measurements, and standards.

MSEL OBJECTIVES

- Foster the Use of Advanced Materials in Commercial Products
- Foster the Development and Implementation of Technologies for Advanced Processing of Materials
- Support the Measurement Base and Standards for Materials Science to Serve the Needs of U.S. Industry

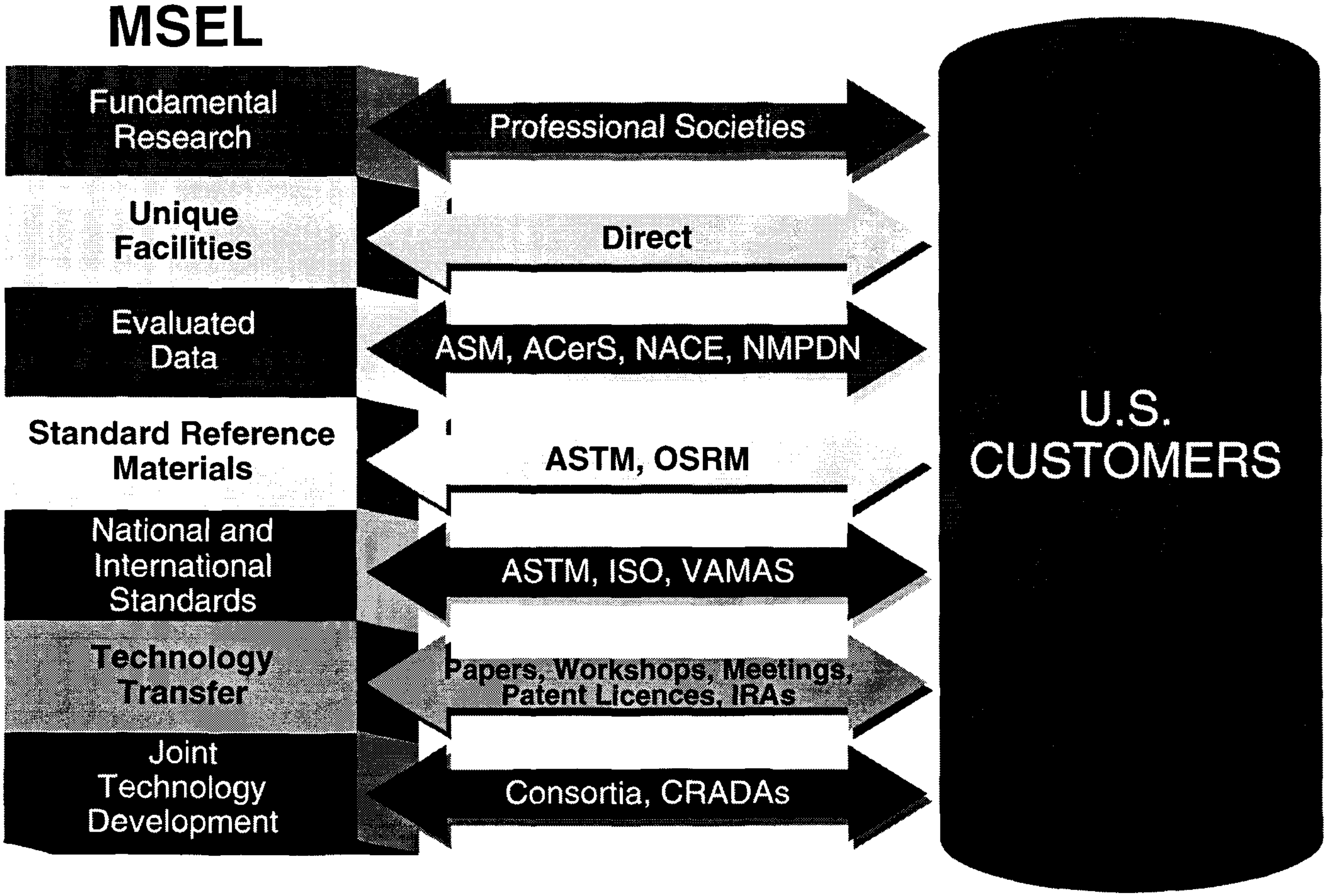
HOW WE ARE ORGANIZED

- By Discipline
  - Ceramics
  - Materials Reliability
  - Metallurgy
  - Polymers
  - Reactor Radiation
- Program Office to Manage Cross-Divisional Programs

MSEL AREAS OF PREEMINENT COMPETENCES

- Processing
  - Metals Solidification
  - Polymer Blends and Composites
  - Process Sensing
- Phase Equilibria
- Mechanical Behavior of Brittle Materials
- Materials Characterization
  - Non-Contact Sensing
  - Ceramic Powder Characterization
- Materials Data
- Neutron Measurement Methods

MSEL-CUSTOMER INTERACTIONS



MSEL

Fundamental Research

Unique Facilities

Evaluated Data

Standard Reference Materials

National and International Standards

Technology Transfer

Joint Technology Development

Professional Societies

Direct

ASM, ACerS, NACE, NMPDN

ASTM, OSRM

ASTM, ISO, VAMAS

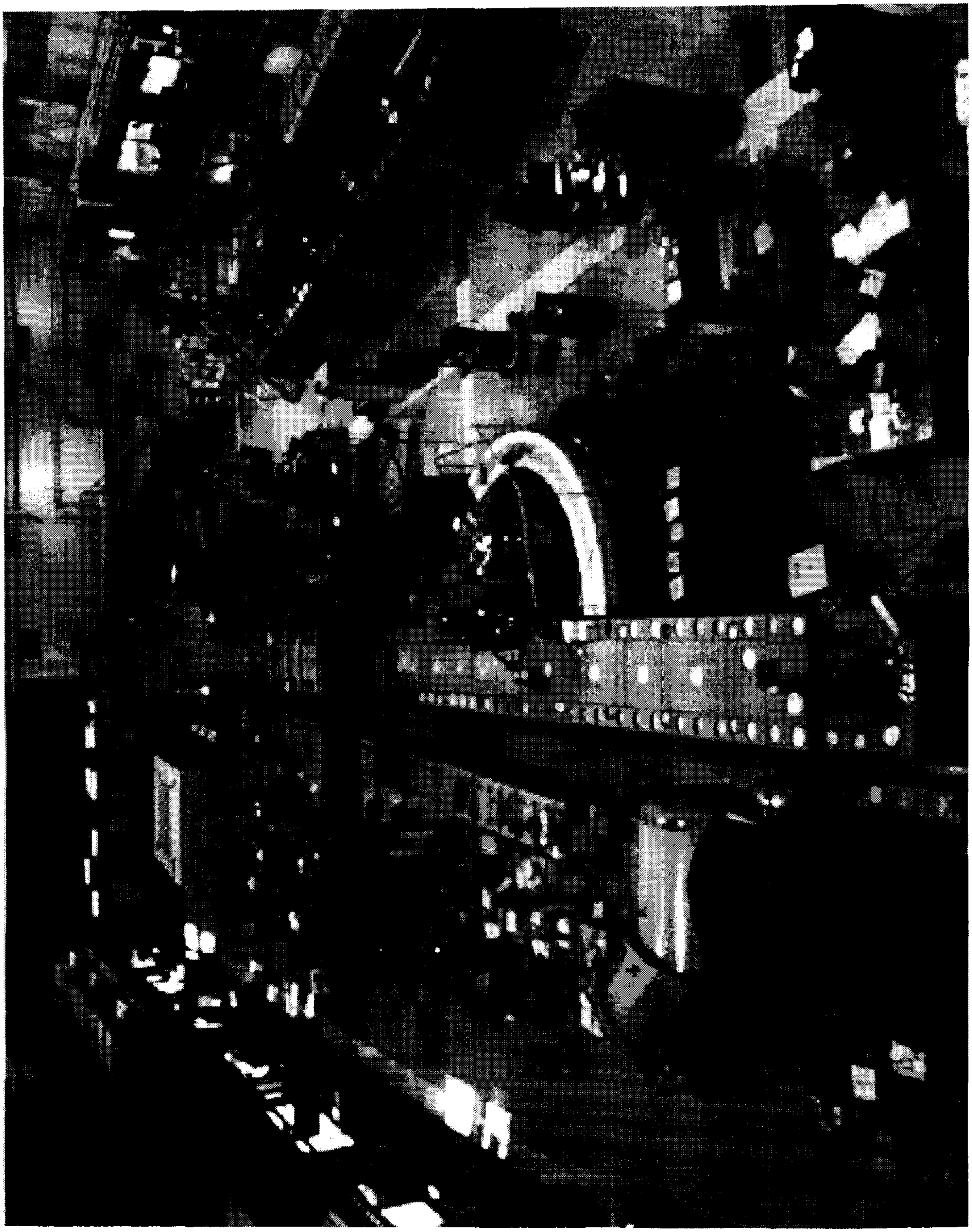
Papers, Workshops, Meetings, Patent Licences, IRAs

Consortia, CRADAs

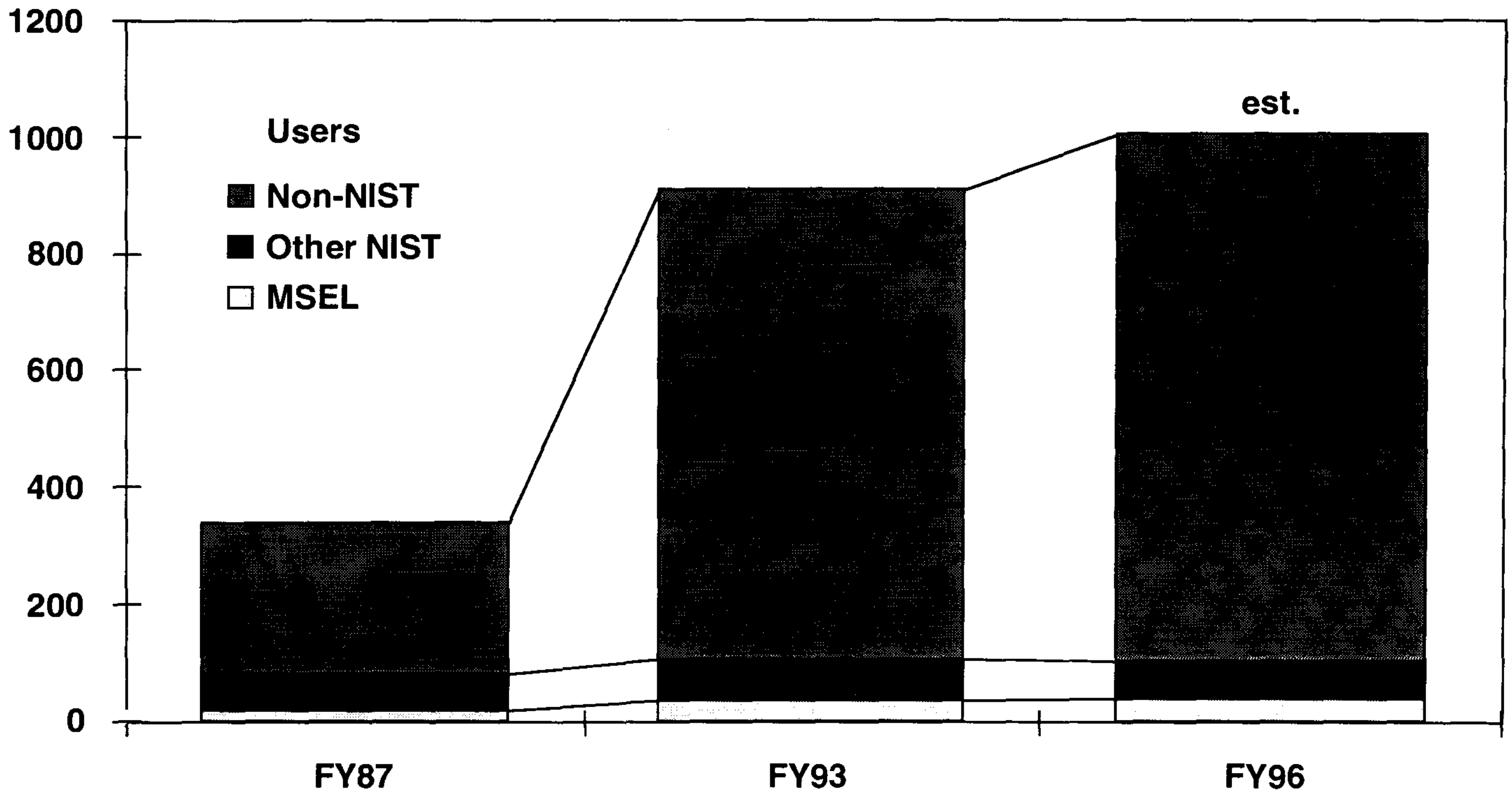
U.S. CUSTOMERS

WHAT WE DON'T DO

- Design Programs for Materials Development
    - Serendipity is followed only with industrial partners
    - The exception to the rule - Dental Materials



FACILITY USE



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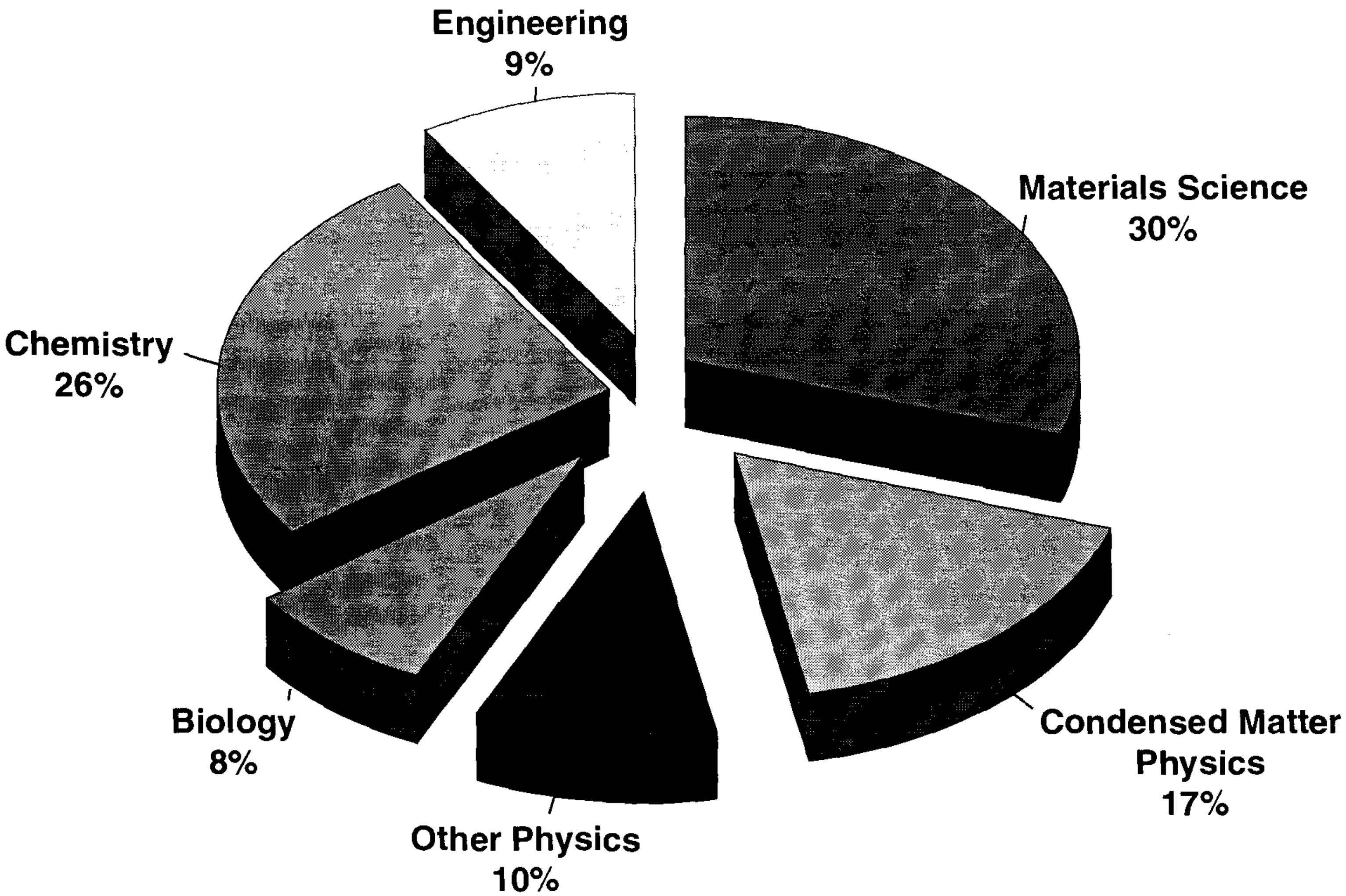
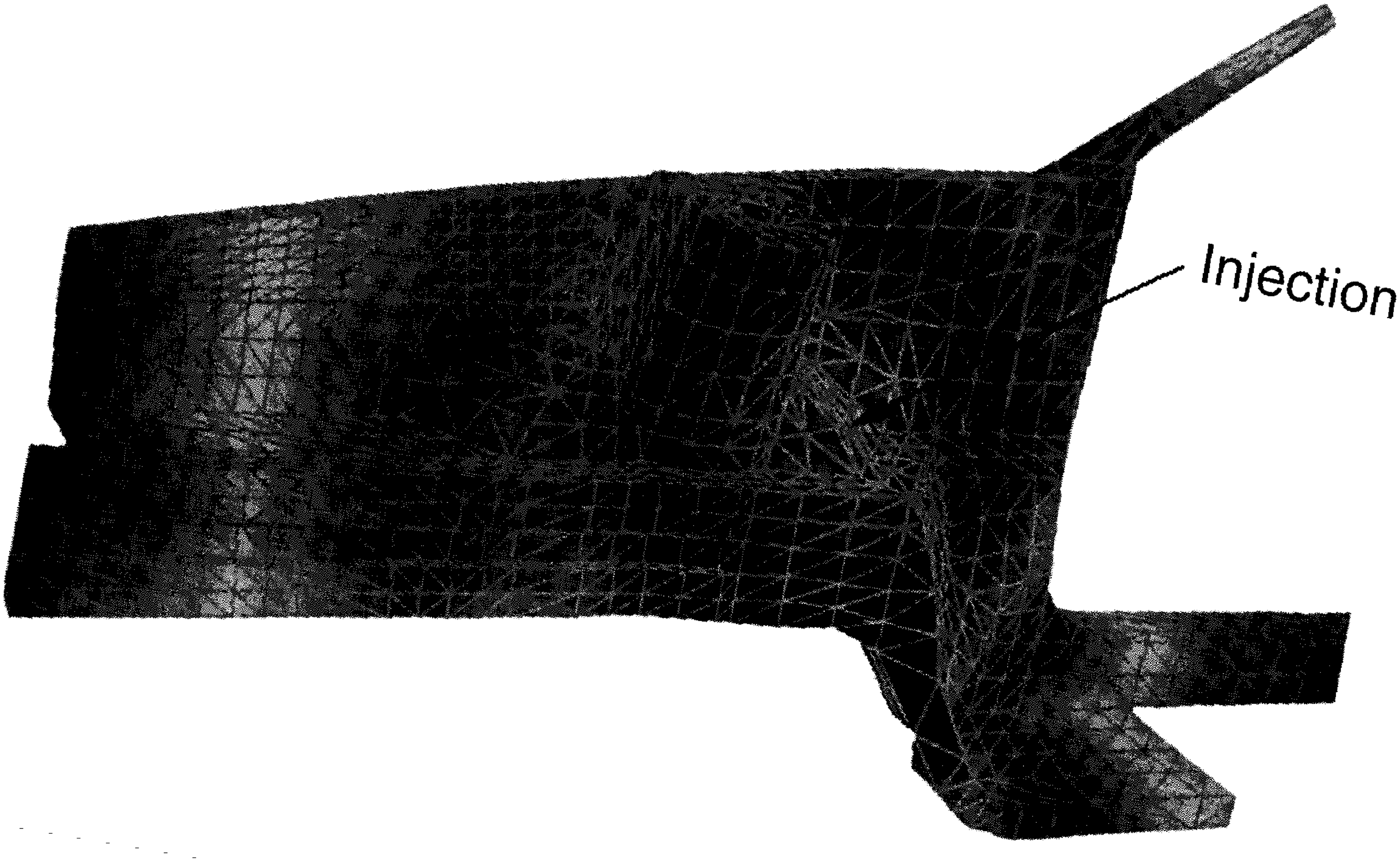


TOTAL USERS = 921 for FY93

MATERIALS SCIENCE AND ENGINEERING LABORATORY PROGRAMS ON TECHNOLOGY DEVELOPMENT

- CRADAs

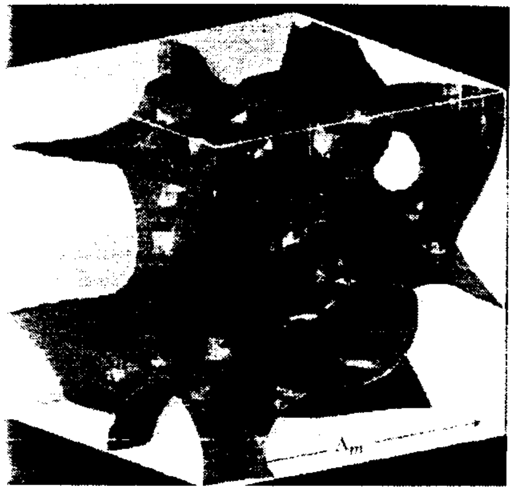
  Examples: Welding
  Polymer Composites
  Metal Casting


Injection

CONSORTIA WITH INDUSTRY: COLLABORATION FOR COMMERCIAL SUCCESS

- Polymer Blends
- Ceramic Machining
- Casting of Aerospace Alloys

NIST/INDUSTRY CONSORTIUM ON POLYMER BLENDS/ALLOYS



ORIGINS:

- Planning Workshops on April 20, 1992 at NIST

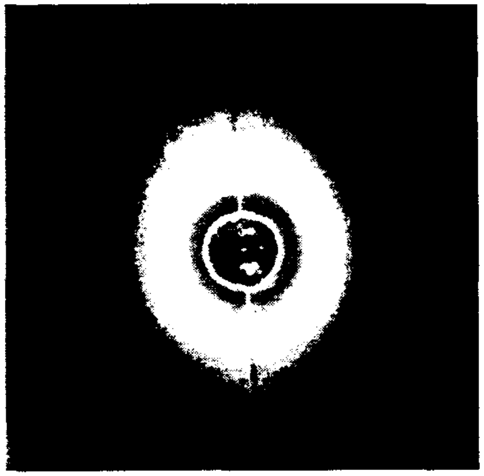
PERIOD OF OPERATION:

- February 1993 to February 1997

MEMBERSHIP: (April 1994)

- Armstrong World Industry
  Aristech Chemical Corp.
  Goodyear Tire and Rubber Corp.
  Raychem Co.
  Rohm & Haas
  Sandia National Lab.
  3M Co.

NIST/INDUSTRY CONSORTIUM ON POLYMER BLENDS/ALLOYS



GOALS:

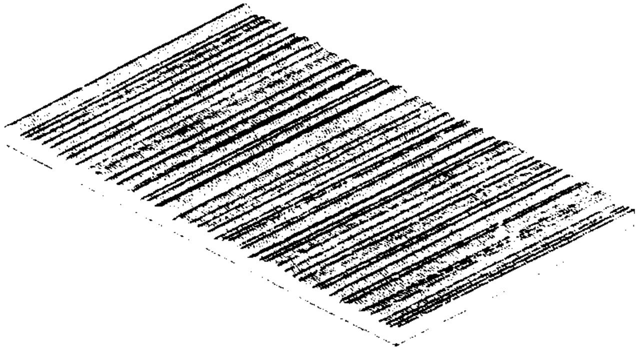
- To Improve the New-Product Development Cycle
- To Improve the Process Design of Polymer Blends/Alloys

APPROACH:

- Thermodynamics and Kinetics of Phase Behavior
- Compatibilization and Interfacial Modification
- On-Line Structure Characterization Instrumentation and Technique for Mixing/Extrusion
- Sensor Development

NIST CERAMIC MACHINING CONSORTIUM

Industry/Government/Universities

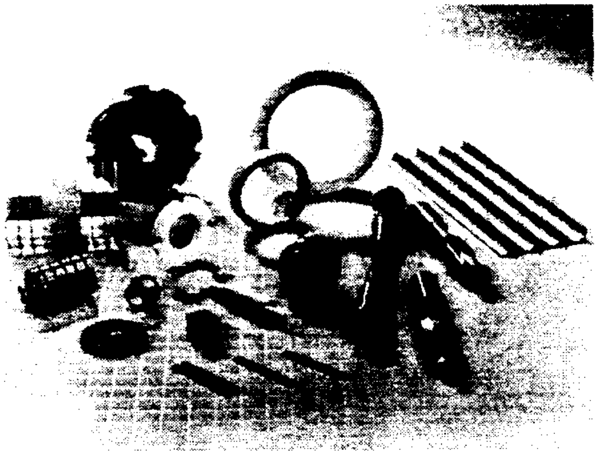


MISSION:
- Assist U.S. industry, through a joint research program, in the development of precision machining technology for the manufacture of reliable and cost-effective products made from advanced ceramics.

GOALS:
- Provide measurement methods, data, and mechanistic information needed by industry for the development of innovative methods for machining of advanced ceramics.

MEMBERS:
- Members provide funds, materials, services in-kind, technical advice, and guidance.

NIST CERAMIC MACHINING CONSORTIUM MEMBERS



Ceradyne, Inc.
Cincinnati Milacron, Inc.
Corning, Inc.
Dow Chemical Company
Eaton Corporation
Eonic, Inc.
Ford Motor Company
General Electric Company
General Motors Corporation
Norton Company
SAC International
Stevens Institute of Technology
Texas A&M University
Torrington Company
West Advanced Ceramics
Tower Oil and Technology
University of Maryland
University of Rochester
W. R. Grace & Company

NIST CONSORTIUM ON CASTING OF AEROSPACE ALLOYS

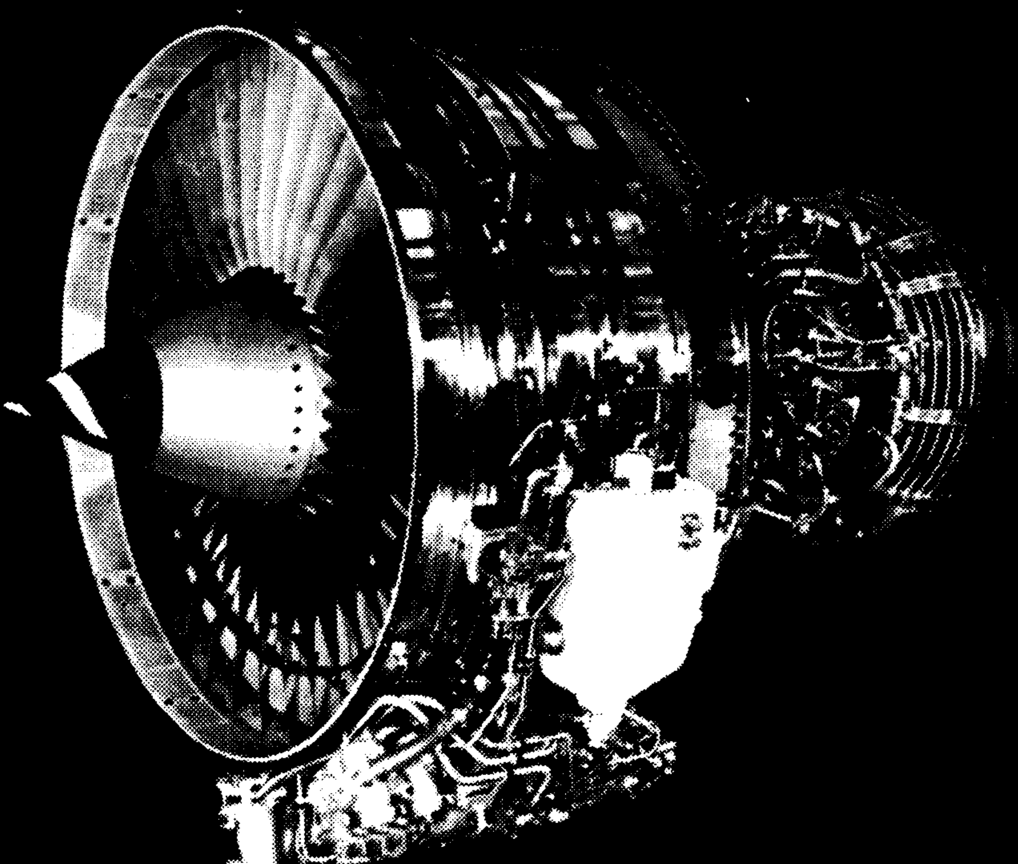
Industry/Government/Universities

GOALS

- To improve the quality and speed of design
- To reduce the reject rate of parts
- To enhance casting product quality

INTELLIGENT PROCESSING O MATERIALS APPROACH

- Process models
- Advanced sensors
- Thermophysical properties data
- Validation and calibration



NIST CONSORTIUM ON CASTING OF AEROSPACE ALLOYS

ORIGINS

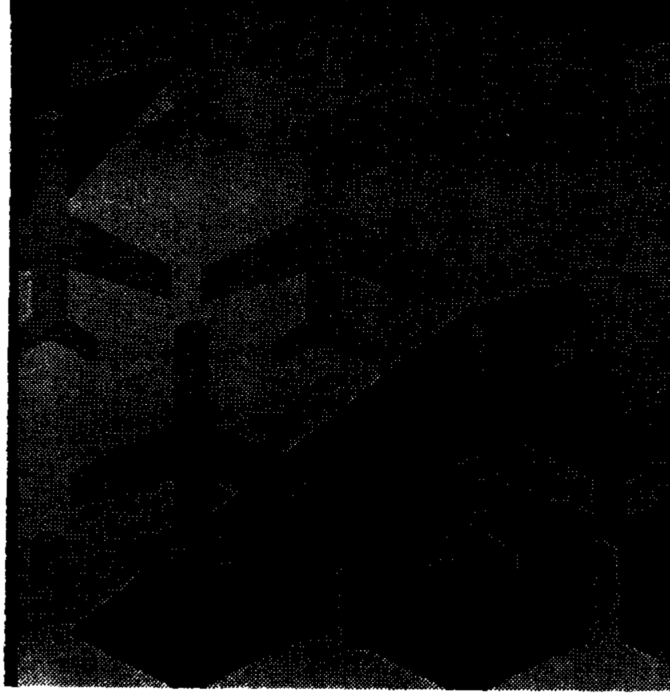
Planning Workshops on:

February 15, 1992 - at Aerospace Industries Association

April 28, 1992 - at NIST

PERIOD OF OPERATION

April, 1993 to April,1997



MEMBERSHIP

(May, 1994)

- AlliedSignal
- Allison Division, GM
- American Foundrymen's Society
- Auburn University
- Bureau of Mines
- Case Western Reserve University
- CINDAS/Purdue University
- GE Aircraft Engines
- Howmet
- Idaho National Engineering Laboratory
- MIT
- NASA
- NIST
- PCC
- Pennsylvania State University
- UES, Inc.
- University of Alabama
- University of Arizona
- University of Illinois
- Worcester Polytechnic Institute

Pending

- Oak Ridge National Laboratory
- Pratt & Whitney
- Air Force Wright Laboratory

BULK ETCHING OF SILICON FOR MICROMACHINING

and

MICROMACHINING OF SUSPENDED STRUCTURES IN SILICON

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Thomas J. Jenkins

and

Victor M. Bright

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Bulk Etching of Silicon for Micromachining

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KEY WORDS: anisotropic etching, microelectromechanical systems (MEMS), micromachining.

PREREQUISITE KNOWLEDGE: The students should be familiar with conventional integrated circuit fabrication. This experiment can be incorporated into an introductory course on solid state sensors. If the students perform the etch, they should have completed a previous laboratory session involving hazardous chemical handling.

OBJECTIVES: This experiment introduces students to the fabrication of microelectromechanical systems (MEMS) and prepares students to conduct more complex experiments in micromachining. The students perform a laboratory experiment to determine the characteristics of anisotropic silicon etching. In particular, students gain experience with anisotropic etches which are peculiar to micromachining, in contrast to their previous experience with isotropic etches used in microelectronic fabrication. After completing this experiment the students will be able to estimate the anisotropic etch rate of different crystal planes of bulk silicon and the resultant etched profiles.

EQUIPMENT AND SUPPLIES: This experiment uses routine equipment found in microelectronic and chemistry laboratories. However if a reflux reactor is not available, it can be purchased or assembled from standard laboratory plastic containers. An example of a handmade reflux reactor is illustrated in Figure 1.

The supplies consist of special etching solutions and silicon substrates for etching. Ethylenediamine, water, and pyrocatechol (EDP) or potassium hydroxide (KOH) is the required etching solution. The students will etch either a test die or a patterned wafer provided by the instructor. The recommended test die consists of a collection of masked features fabricated through the MOS Implementation Service (MOSIS). A suggested layout and the specific MOSIS criteria are described in the Instructor Notes. If MOSIS is not available, a patterned silicon wafer can be used. The wafer specifications are in the Instructor Notes.

PROCEDURE: The experiment is conducted in a sequence of steps. These steps require the students to predict the etch results, prepare the etching solutions, perform an etch, and analyze the results. Also, students should be properly briefed concerning the hazards of the various etching solutions. Adequate protection, such as goggles, gloves, and aprons, as well as proper ventilation, must be used. The etching solutions are extremely corrosive and toxic.

Prediction: The students begin by predicting the resultant etch for each of the open shapes. The predictions consist of: profile, etch time, and final etch depth. The layout, its crystallographic orientation, and etching conditions are provided to the students to enable the calculations.

Etching solution preparation: The students prepare the selected etching solutions. EDP and KOH are the preferred anisotropic etching solutions. A buffered solution of hydrofluoric (HF) acid must also be prepared. Since silicon surfaces will readily form a thin native oxide whenever exposed to room air, the HF is required to strip any native oxide which forms in the regions to be anisotropically etched.

The EDP should be purchased already mixed. However, the KOH will need to be mixed. A solution of 44 grams of KOH crystals in 100 ml of deionized (DI) water will produce reliable results. The HF should be mixed in a 5% solution with DI water.

Etching: Before the test die are etched, the die must be dipped in the HF solution for 10 seconds and then thoroughly rinsed in DI water. After rinsing, the die are immediately placed in the etch apparatus and dipped in the anisotropic etching solution. The die must be handled with Teflon or other types of plastic tweezers. The etching solutions will corrode metal parts. For proper results, the EDP and the KOH must be maintained near 95°C and 85°C, respectively. While etching, both etching solutions must be gently stirred.

After etching the die are removed from the etch apparatus, and thoroughly rinsed in DI water. The etch time should be recorded to determine the etch rate during analysis.

The die are then dried by removing the excess water with a lint-free tissue and left to dry in the air. Also, the die can be placed in an oven for a few minutes at 100°C.

Examination and Analysis: The students examine the test die to determine the characteristics of the etched pits. The test die can be examined with an optical microscope to determine the etch depth and profile. If a profilometer is available, the students can use this instrument to measure the profile, as well. The students should correlate the pit characteristics with the lateral dimensions and crystallographic orientation of the masked regions used to pattern the pits. Significant deviations from their predicted results should be explained as part of the laboratory report.

SAMPLE DATA SHEETS: Self-Evident.

INSTRUCTOR NOTES: An introduction to the emerging field of MEMS and micromachining should combine hands-on laboratory experiments with class room lectures. The experiment described in this paper complements a discussion of bulk silicon micromachining as it applies to fabrication of MEMS. A CMOS-compatible process may be used for this purpose.

Suggested Lecture Materials: To obtain the most benefit from this experiment, the prerequisite knowledge in microelectronics should be supplemented with an associated lecture covering the following topics: motivation for MEMS, silicon crystal structure, the properties of anisotropic etching, the influence of crystal orientation with respect to the device geometry, and the electronic materials compatibility issues.

Micromachining is a developing field which promises to reduce device size and weight in many applications [1], [2]. Micromachining has already moved into commercial production in the area of accelerometers. For example, substantial markets are developing as micromachined accelerometers are incorporated in automotive air bag systems. Since micromachining is closely related to microelectronic fabrication techniques, it makes sense to introduce the topic in microelectronic courses. The ability to incorporate micromechanical structures with integrated circuits enables the development of improved sensors and other instrumentation.

MEMS can be fabricated using many different processes. One such process is bulk silicon anisotropic etching. This process uses the unique crystallographic properties of silicon to form cavities and suspended structures. These mechanical features are fabricated in the same bulk material which is used to fabricate integrated circuits. For example, conventional CMOS processing can be combined with bulk silicon micromachining [3].

Miller indices are used to define crystal planes. The Miller indices and standard orientations in silicon wafers are illustrated in Figure 2. Figure 3 should be photocopied onto heavy paper, cut-out, and then folded to produce a 14-sided model of a silicon crystal. This model can be used to illustrate the crystal planes.

In silicon, the atoms are spaced more closely in some crystal directions than others. In particular, the <111> direction has the least space between atoms, while the <100> direction has the most space between atoms. Some chemicals etch along these crystal directions at different rates, so such etching solutions are called anisotropic. For example, EDP will etch in the <100> direction 35 times faster than in the <111> direction at 115°C, while for KOH the ratio can be 400:1 at 85°C for those same directions [4]. This selectivity permits the use of the {111} planes as etch stops when etching silicon wafers whose faces are {100} oriented. Figure 4 shows how wafers are given a flat to indicate the orientation of the various crystal planes. Standard CMOS circuits are fabricated using {100} wafers, so an anisotropic etch will result in the profile shown in Figure 5. This figure also compares the anisotropic etch with the isotropic etch normally used in microelectronic circuit fabrication.

By defining a rectangular mask opening aligned to the {110}-oriented wafer flat, a pit can be etched into the wafer. The etch proceeds in all directions, but fastest in the <100> direction. At the edges of the pit, where {111} planes intersect the sides of the mask opening, the etching solution cannot get to a faster etching surface, so those planes are gradually exposed as the etch proceeds. The etch virtually stops when only those four {111} planes are left exposed, which leaves a pyramidal pit in the wafer. Since there is some slow etching in the <111> direction, there will be a slight undercutting of the mask edges. To reduce this undercutting, a heavy p-type diffusion can be placed around the mask opening which further slows down the etch process [3].

Reflux Reactor and Other Supplies: The reflux reactor, which is shown in Figure 1, was fabricated from a plastic beaker (250-ml) with a plastic cover. A slightly smaller beaker was placed inside to suspend a two-inch fluoroware wafer holder. The bottom of the smaller beaker was perforated with four 3/4-inch holes. Also, the wafer holder was perforated with several dozen small holes which are large enough to permit the free flowing of the etching solution, but small enough to prevent the test die from falling through the basket. This smaller beaker should rest inside the larger beaker with enough space so as not to impede the stir bar action. The cover has a small access hole in the center to allow the insertion of a thermometer tube. This reactor was fabricated with plastic parts, but glass is also acceptable. Metal components are not recommended.

The test die should contain some adaptation of the features depicted in Figure 6, which is a simple etch gauge. The gauge was fabricated through MOSIS. If the design is submitted to MOSIS, the Orbit SCNA_MEMS or SCPE_MEMS process must be used [3]. The suggested features consist of square and rectangular patterns of various dimensions. This collection of patterns will permit the students to observe the effects of various dimensions on the etch time and pit profile. Also, the students will be able to observe the differences between KOH and EDP etching solutions.

If the test die can not be obtained through MOSIS, a suitable alternative can be fabricated in a microelectronics laboratory. The initial silicon wafer should be {100}-oriented. The patterns to be etched should be masked with silicon dioxide. No specific oxidation schedule needs to be followed; however, the oxide should be pin-hole free and at least 210–nm thick [5]. The dimensions of the patterned features should range from 20 to 100 micrometers. Using these feature sizes, the students will be able to make adequate observations to determine the etch rates. An example wafer processing technique can be found in [6].

Anticipated Results: The etch should be judiciously timed so that some features are completely etched while others are not. This will allow estimates of etch rate based upon the size of the largest completely etched pit. Also, an incompletely etched pit can be measured to determine the etch depth, and thus, the etch rate. Students should use the method shown in Figure 7 to calculate the etch depth. The students should correctly observe that the etch rate is the same for all features; however, the final depth of the etched pit is determined by the length of the shortest side of the rectangular feature. The etch process is a self-terminating process which is determined by the geometry of the layout. Hence, the students should realize the utility of anisotropic etching solutions in the fabrication of MEMS.

Upon conclusion of the experiment, the students should observe the various states of completion of the etched pits as the dimensions of the masked features vary. An example of etch results using EDP is shown in Figure 8. The etch duration was 30 minutes, and the etch rate was about 0.54 micrometers per minute in the <100> direction. The smallest completely etched pit was 23x23 micrometers. Figure 9 depicts an example of etch results using KOH after 2.5 minutes of etching. For this case, the smallest completely etched pit was 19x19 micrometers, and the etch rate was 5.4 micrometers per minute.

Suggested Follow-on Experiments: The knowledge gained in this experiment will prepare the students for the subsequent experiment where they will fabricate suspended structures.

SOURCES OF SUPPLY: The routine chemical supplies and KOH are available from Sigma Chemical Corp., P.O. Box 14508, St. Louis, MO 63178. The EDP can be purchased already mixed from Transene Co., Inc., US Rt. 1, Rowley, MA 01969.

REFERENCES:

1. J. Bryzek, K. Petersen, and W. McCulley, "Micromachines on the march," *I. E. E. E. Spectrum*, pp. 20-31, May 1994.

2. G. Stix, "Micron machinations," *Scientific American*, pp. 107-117, Nov 1992.

3. J. Marshall, M. Gaitan, M. Zaghloul, D. Novotny, V. Tryee, J.-I. Pi, C. Piñá, and W. Hansford, "Realizing suspended structure on chips fabricated by CMOS foundry processes through the MOSIS service," NISTIR 5402, U. S. Department of Commerce, National Institute of Standards and Technology, June 1994.

4. K. E. Petersen, "Silicon as a mechanical material," *Proc. I. E. E. E.*, vol. 70, pp. 420-457, May 1982.

5. L. Ristic, ed., *Sensor Technology and Devices*, Artech House, Norwood, MA, 1994.

6. T. Jenkins and E. Kolesar, "Laboratory experiments in integrated circuit fabrication," *NASA Conference Publication 3201, National Educators' Workshop: Update 1992, Standard Experiments in Engineering Materials Science and Technology*, National Aeronautics and Space Administration Office of Management, Scientific and Technical Information Program, pp. 235-270, 1993.

7. S. M. Sze, *Semiconductor Devices - Physics and Technology*, John Wiley & Sons, New York, 1985.

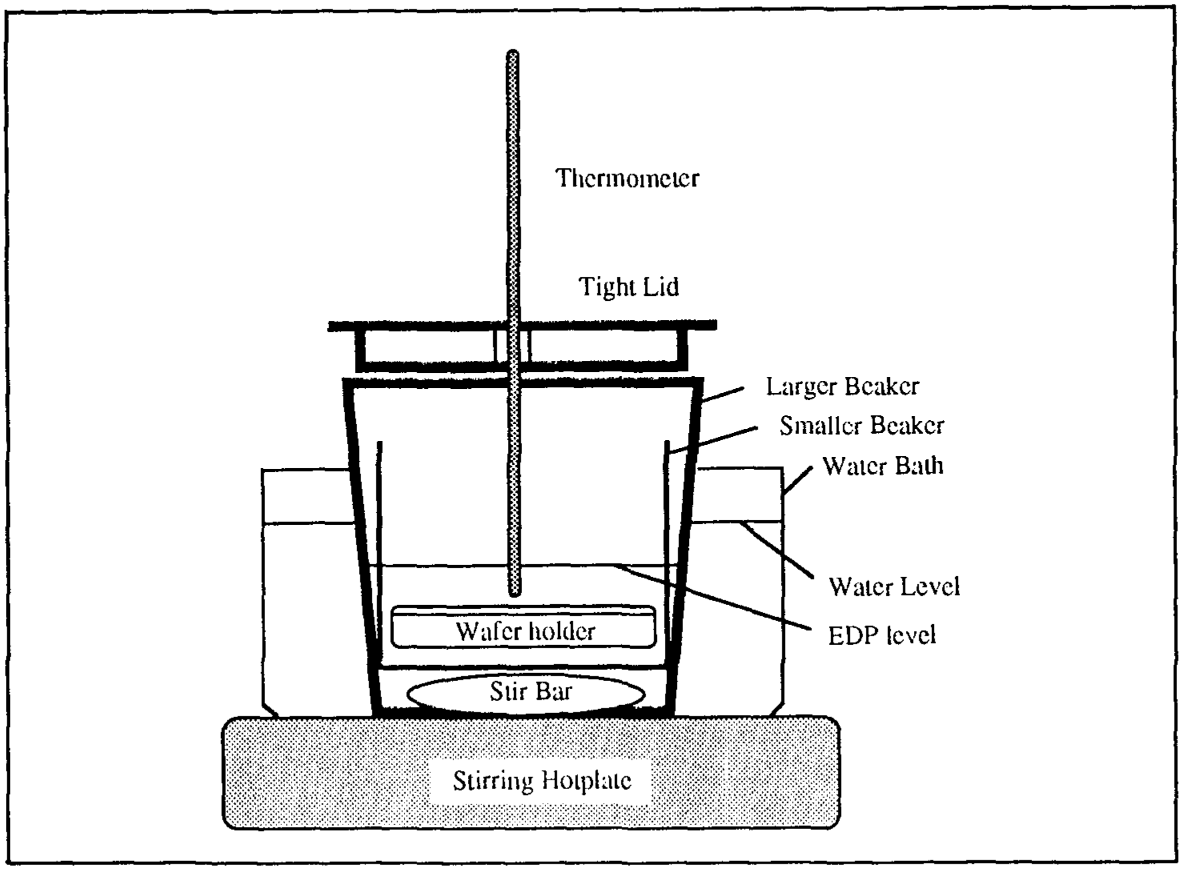

Figure 1. Diagram of lab-built reflux reactor.

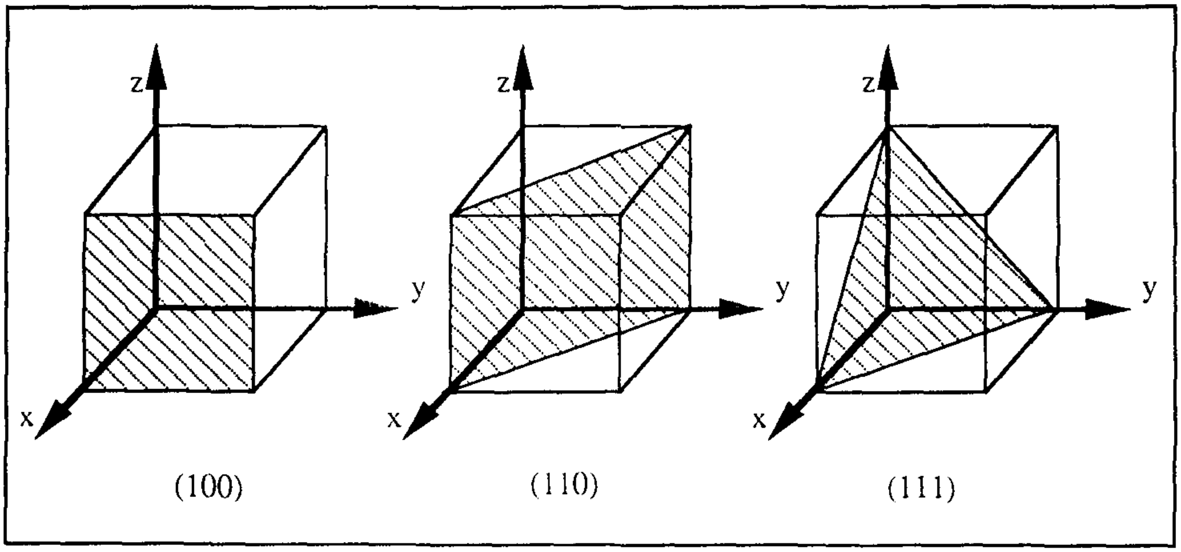

Figure 2. Miller indices of some important planes in cubic crystals [7].

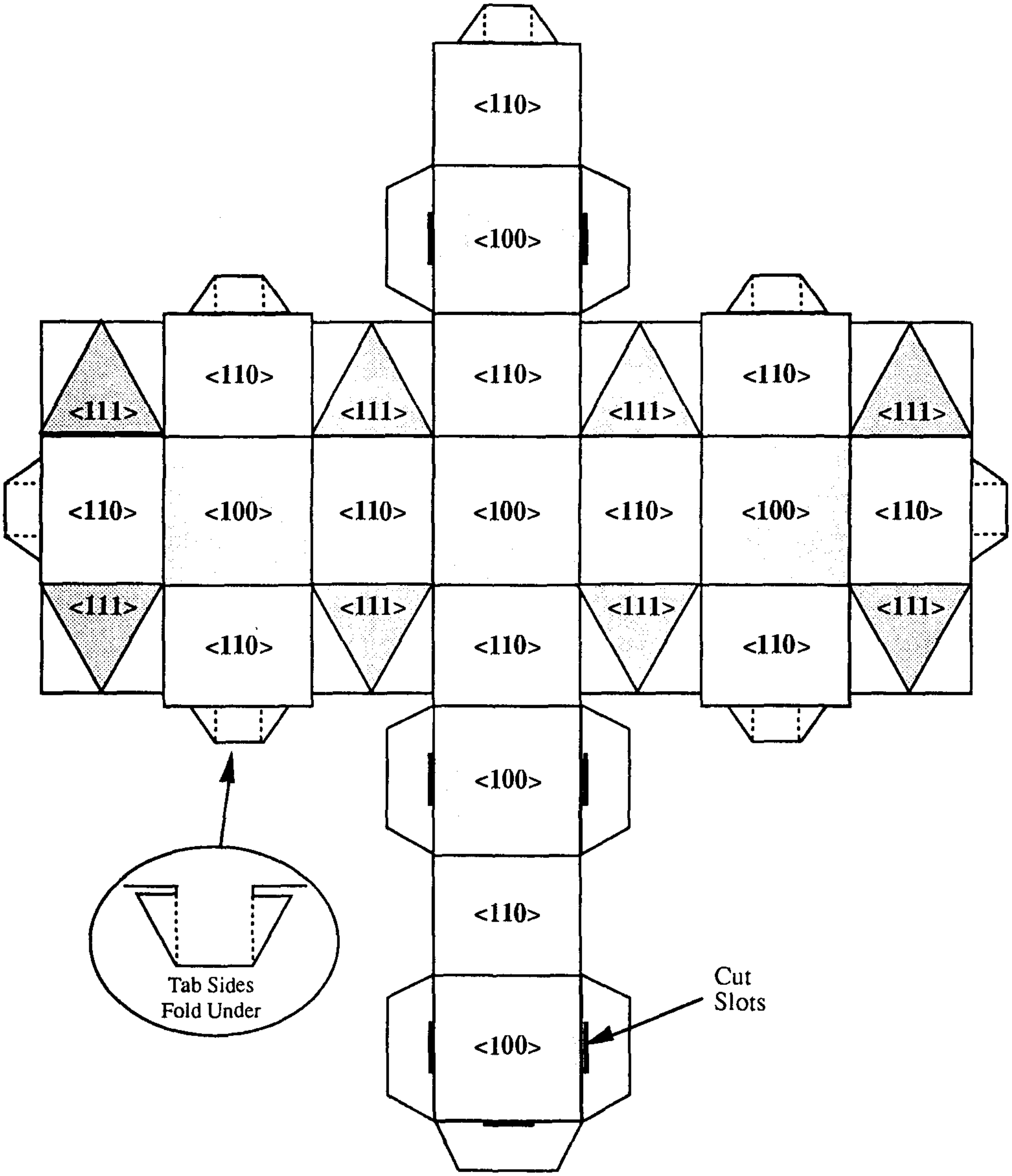

Figure 3. Fourteen-sided crystal model cutout.

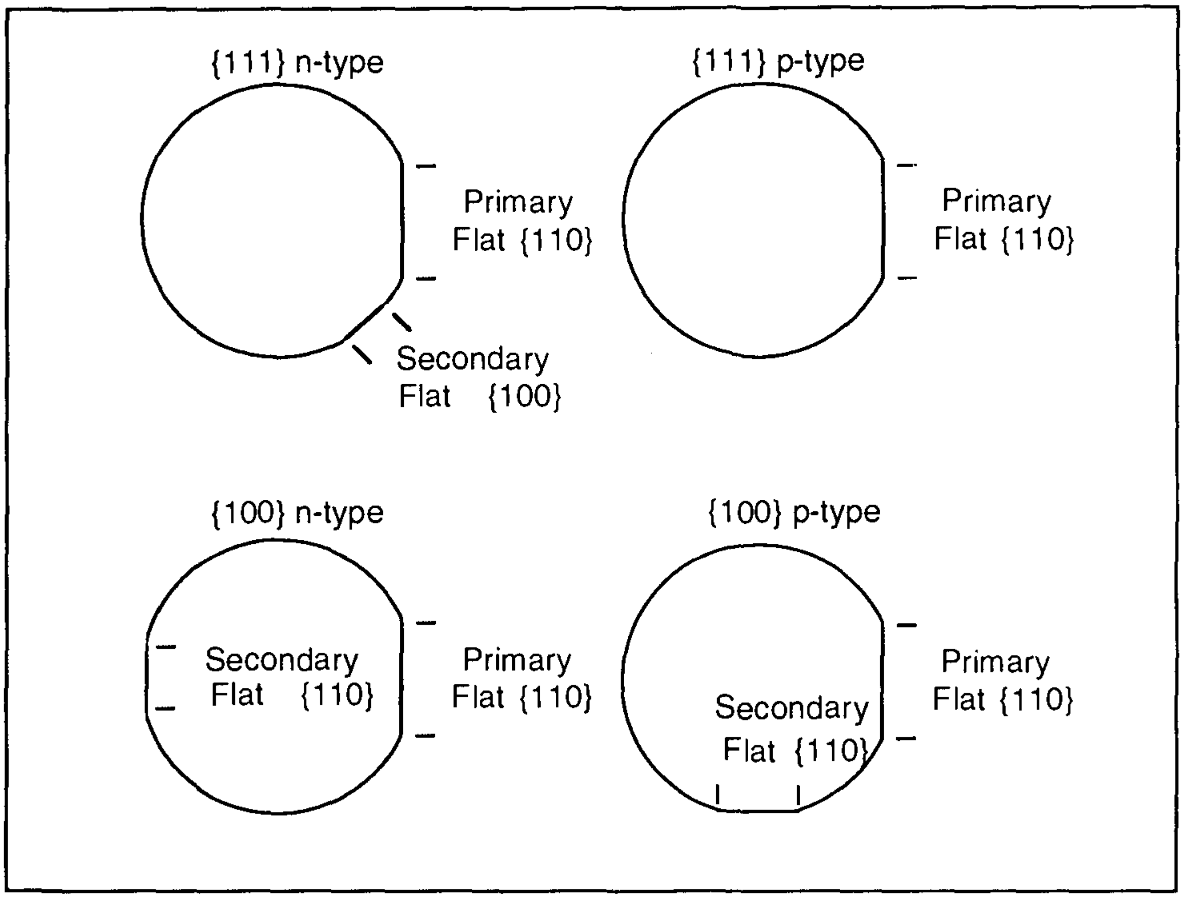

Figure 4. Identifying flats of a silicon wafer [after 7].

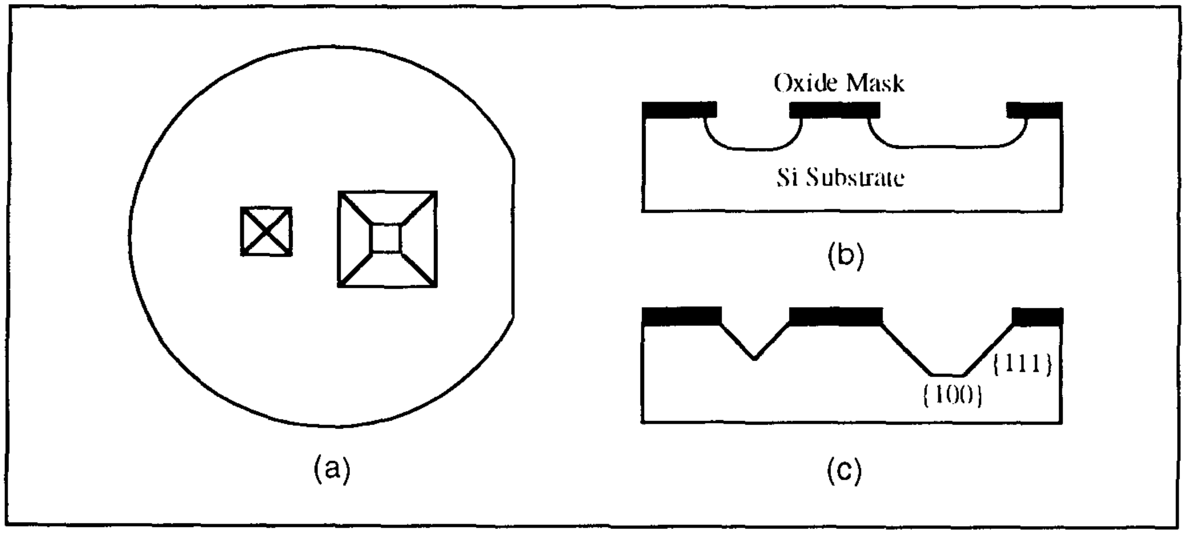

Figure 5. Illustration of differences between isotropic and anisotropic etch of {100} silicon wafer with similar masks. (a) Mask alignment parallel to the primary wafer flat. (b) Profile of isotropic etch. (c) Profile of anisotropic etch showing crystal plane directions.

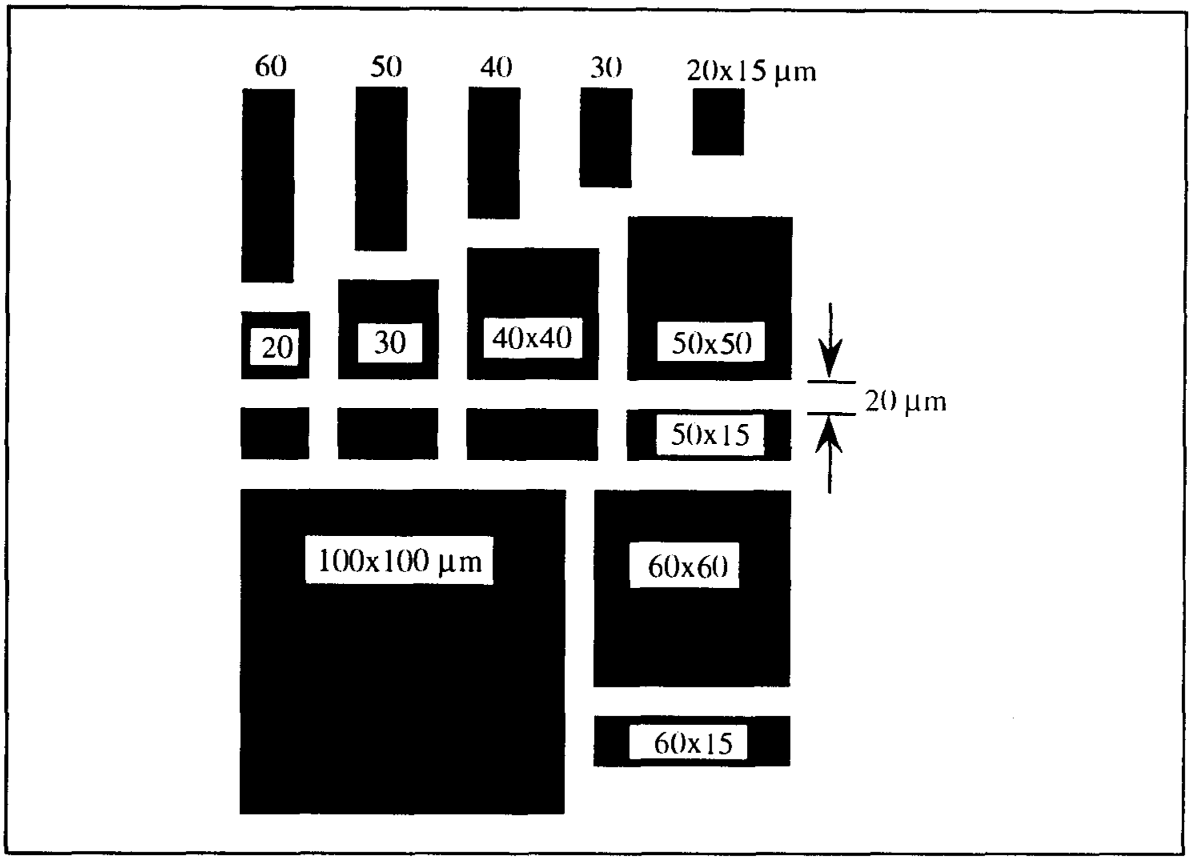

Figure 6. Suggested layout for test die. The minimum spacing between boxes is 20 µm.

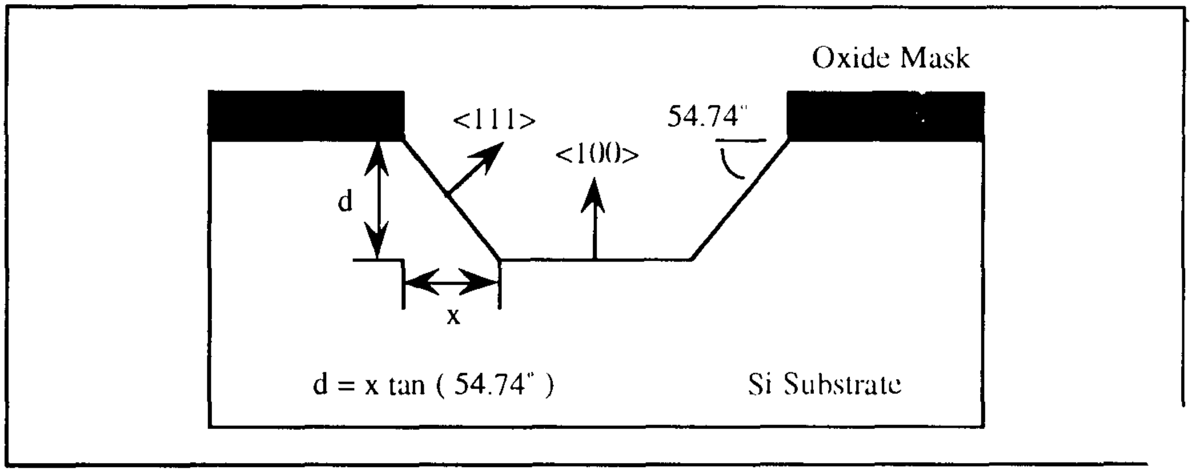

Figure 7. Profile of an incompletely etched pit for a masked {100} surface orientation.

Figure 8. An example of etch results using EDP for 30 minutes. The largest completely etched pit is 23x23 µm.

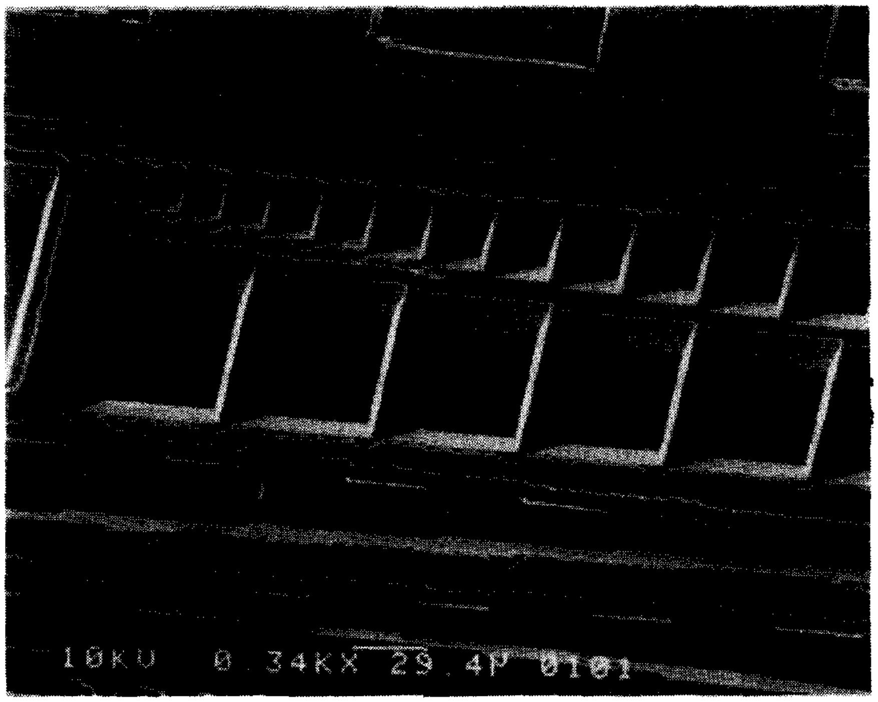

Figure 9. An example of etch results using KOH for 2.5 minutes. The largest completely etched pit is 19x19 µm.

have been mistakenly predicted. Referencing Figures 8 and 9, the following results should be observed:

Pattern (a) is a repeat of the pattern used for the prerequisite experiment in bulk silicon etching [1]. Students should correctly predict that it will etch into a square pyramidal pit with no suspended features.

Pattern (b) is a square oriented at 45° to the primary wafer flat. It will etch into a square pit with overhanging glass at the corners.

Pattern (c) will etch into two unconnected pits. The upper pit will have two released cantilevers and an overhang at the top. The bottom pit will have a single cantilever.

Pattern (d) will result in the trampoline structure over a single pit.

Pattern (e) will initially etch into a square torus supporting the square of glass in the center on a pillar of unetched silicon. As the etch proceeds the pillar will get narrower until the glass square is released and rinsed away in the etching solution. The pit will continue to etch into a square pyramidal pit like pattern (a).

Pattern (f) will result in a cantilever suspended over a square pit similar to the cantilevers depicted in Figures 3 and 4.

Pattern (g) will result in a suspended square of glass supported by two arms over a square pit.

Pattern (h) is similar to pattern (g), but it will result in four unconnected pits with no suspended structure. In this case, the etch will terminate before the pits can join together.

The students should also conclude that the full pit depth does not have to be reached in order to fully release the structures. The time actually needed for full release depends on the dimensions of the suspended structures and open areas.

Suggested Follow-on Experiments: The students should be prepared to design and fabricate actual devices. For example, they could design, fabricate, and test some of the devices in [5], such as the diving board cantilever (Figures 3 and 4) and the infrared point source (Figure 5).

SOURCES OF SUPPLY: The routine chemical supplies and KOH are available from Sigma Chemical Corp., P.O. Box 14508, St. Louis, MO 63178. The EDP can be purchased already mixed from Transene Co., Inc., US Rt. 1, Rowley, MA 01969.

REFERENCES:

1. T. Jenkins, J. Comtois, and V. Bright, "Bulk etching of silicon for micromachining," *National Educators' Workshop: Update 1992, Standard Experiments in Engineering Materials Science and Technology, NASA CP-* , Nov. 1994.

2. J. Bryzek, K. Petersen, and W. McCulley, "Micromachines on the march," *I. E. E. E. Spectrum*, pp. 20-31, May 1994.

3. G. Stix, "Micron machinations," *Scientific American*, pp. 107-117, Nov. 1992.

4. M. Mehregany, "Micromechanical systems," *Circuits and Devices*, pp. 14-22, July 1993.

5. J. Marshall, M. Gaitan, M. Zaghloul, D. Novotny, V. Tryee, J.-I. Pi, C. Piñá, and W. Hansford, "Realizing suspended structure on chips fabricated by CMOS foundry processes through the MOSIS service," NISTIR 5402, U. S. Department of Commerce, National Institute of Standards and Technology, June 1994.

6. J. Y.-C. Chang, A. A. Abidi, M. Gaitan, "Large suspended inductors on silicon and their use in a 2 μm CMOS RF amplifier," *I.E.E.E. Electron Device Letters*, vol. 14, pp. 246-248, May 1993.

7. J. Marshall, M. Parameswaran, M. Zaghloul, M. Gaitan, "High-level CAD melds micromachined devices with foundries," *Circuits and Devices*, pp. 10-17, Nov. 1992.

8. T. Jenkins and E. Kolesar, "Laboratory experiments in integrated circuit fabrication," *NASA Conference Publication 3201, National Educators' Workshop: Update 1992, Standard Experiments in Engineering Materials Science and Technology*, National Aeronautics and Space Administration Office of Management, Scientific and Technical Information Program, pp. 235-270, 1993.

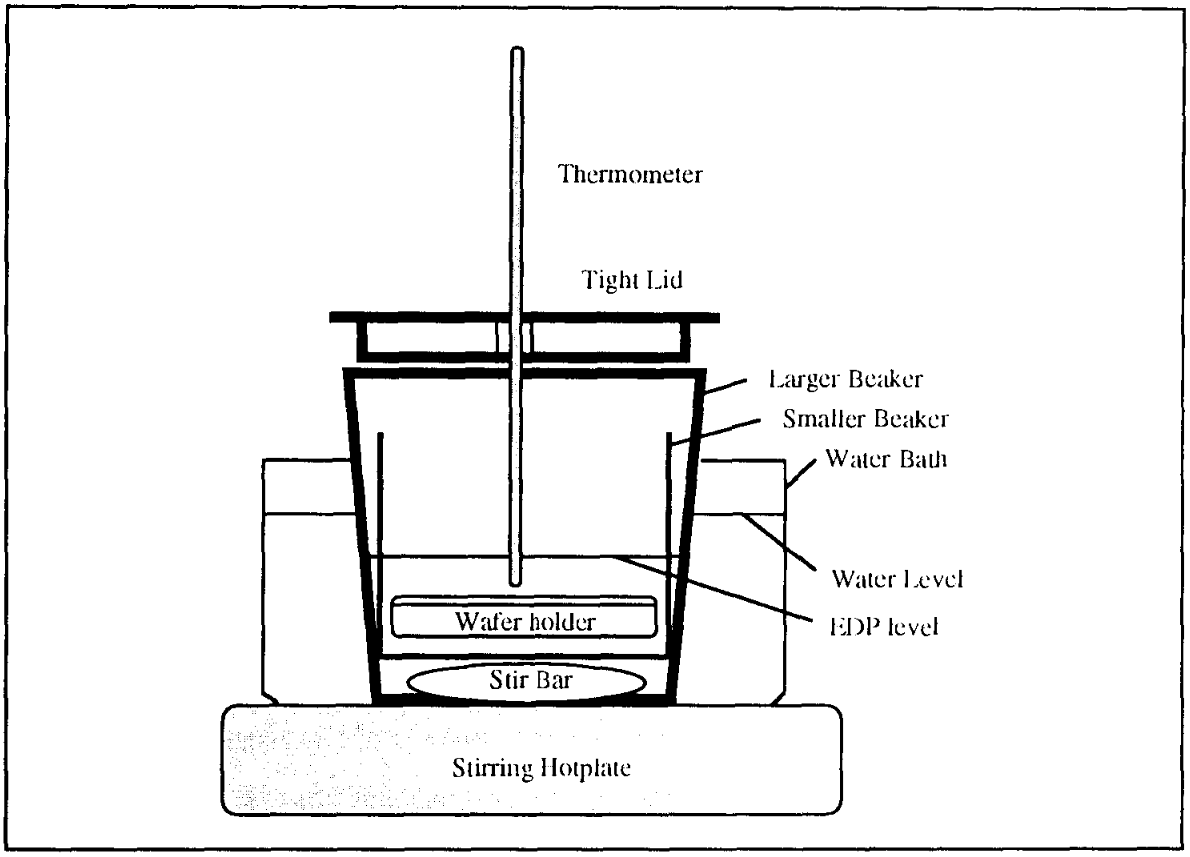

Figure 1. Diagram of lab-built reflux reactor [1].

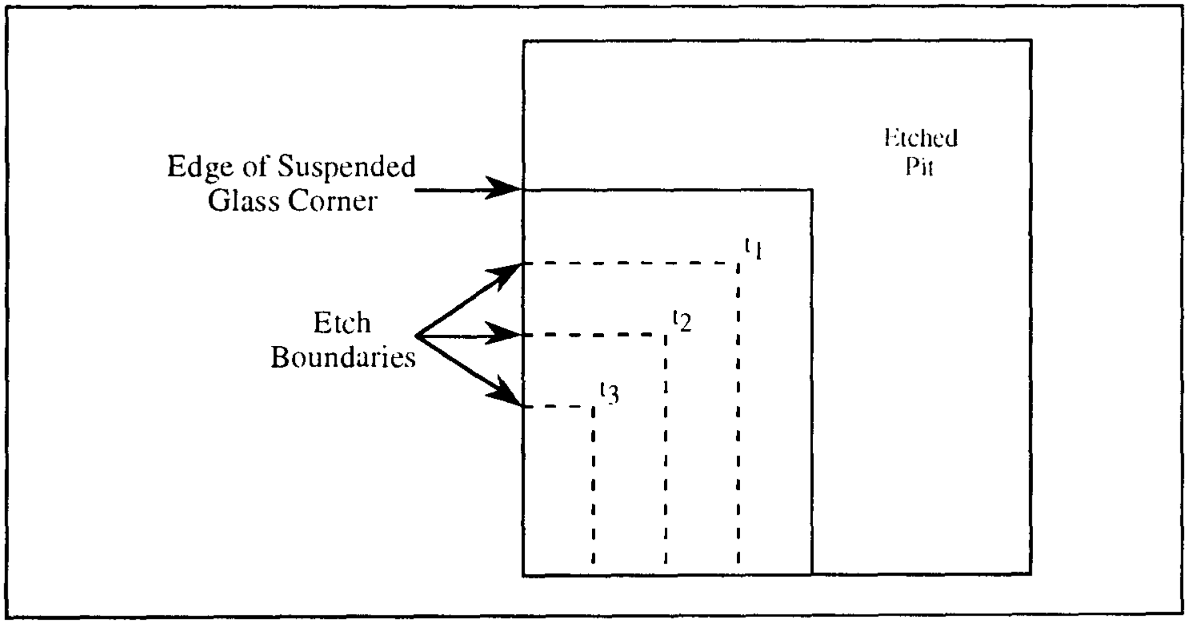

Figure 2. Example of etch progression as it undercuts the edges of a suspended glass corner. The dashed lines illustrate the extent of the etched pit at successive times (t).

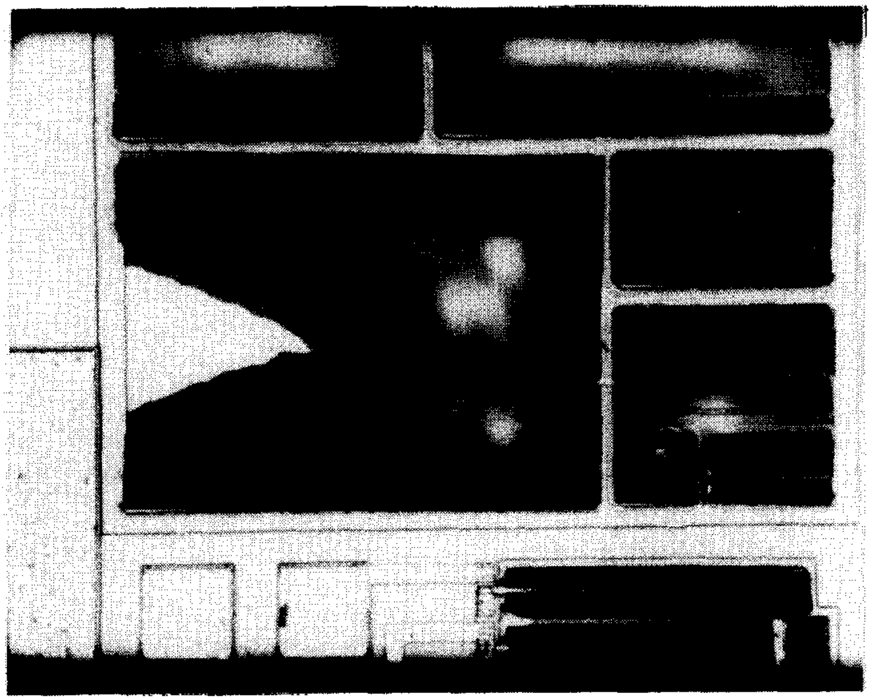

Figure 3. Undercut of glass cantilevers after 135 minutes in EDP at 85°C. The largest cantilever is 135x250 µm.

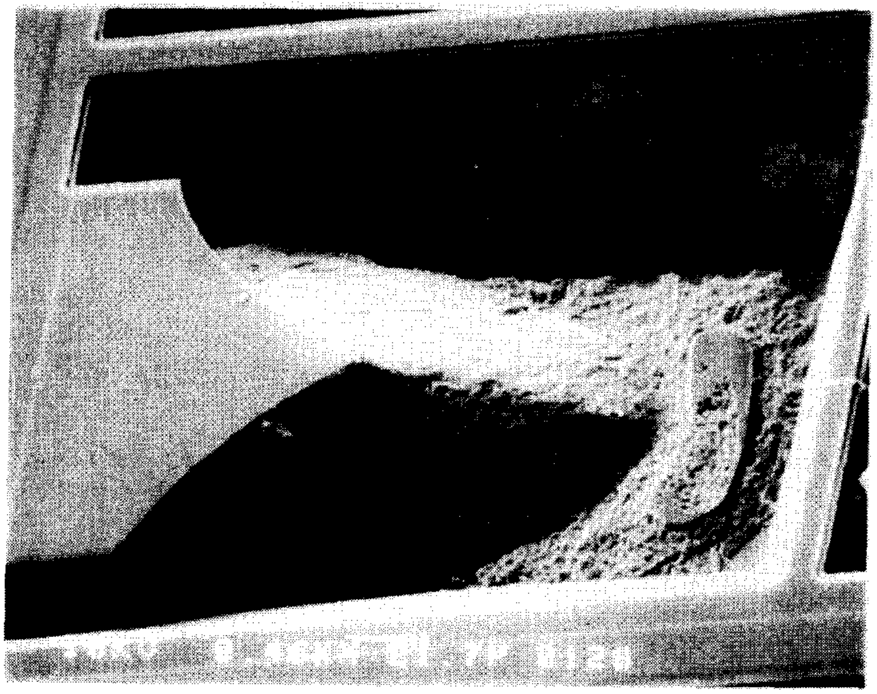

Figure 4. 135x250 µm glass cantilever partially etched in EDP for 135 minutes at 85°C. The cantilever was broken off to reveal the progress of the etch. The pit is 192x266 µm.

Figure 5. Suspended resistive heater using a trampoline structure. The center of the trampoline is 85x85 μm and the pit is 180x180 μm.

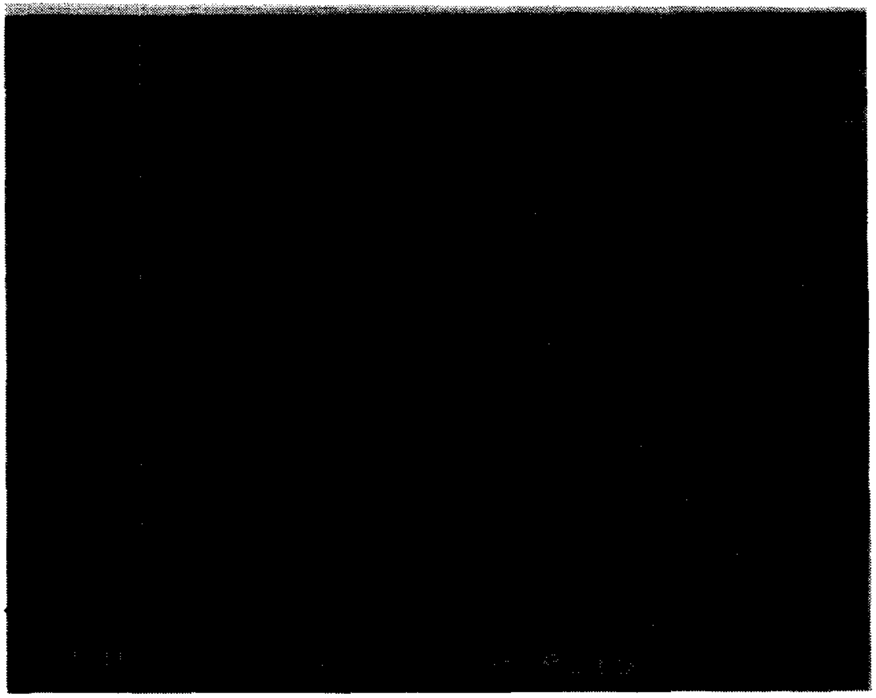

Figure 6. Suspended resistive heater using a cantilever structure with support arms at 45° angle. The cantilever is 95x150 μm and the pit is 180x200 μm.

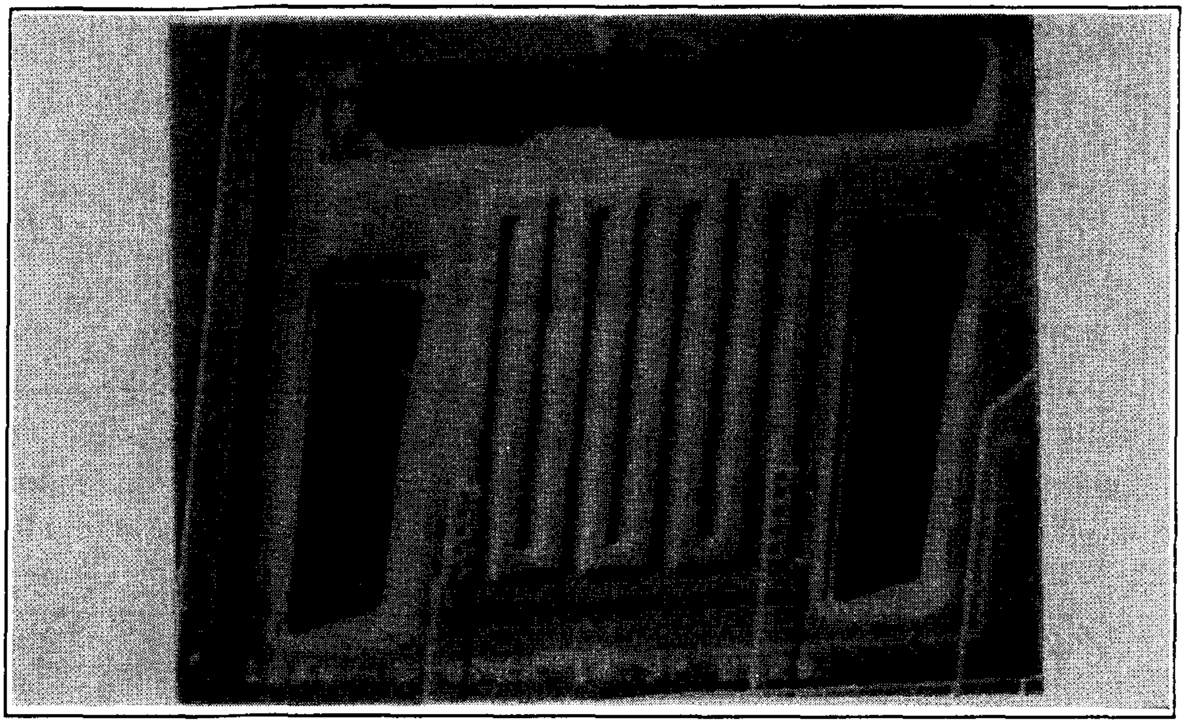

Figure 7. Result of placing support arms at 90°. The 95x103 μm cantilever is not released.

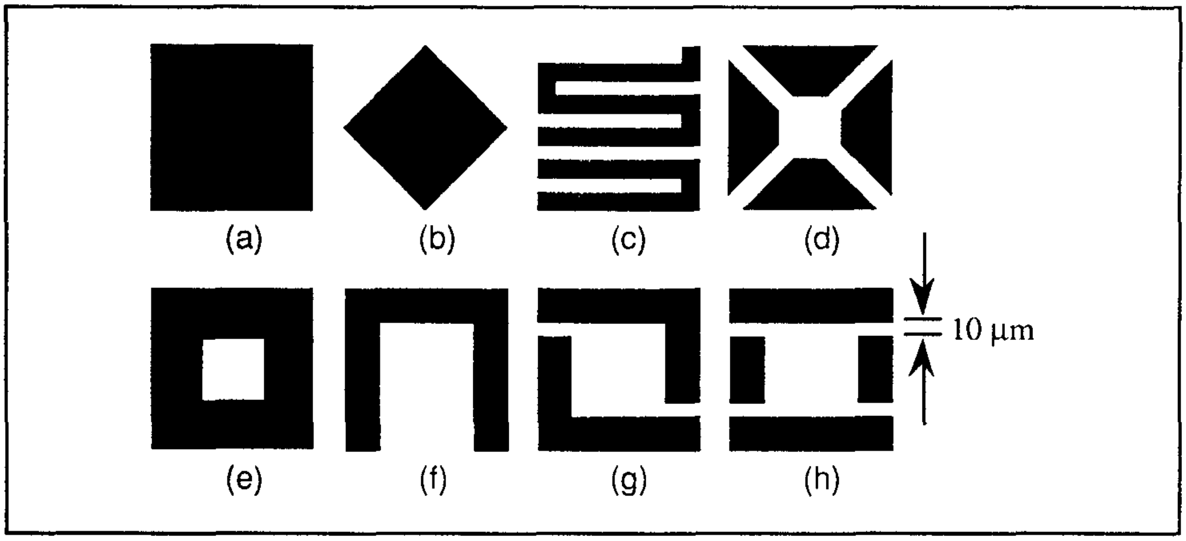

Figure 8. Suggested layout of test pattern masks. Minimum spacing between mask openings (indicated by black) is 10 μm. The overall outer dimension of each test pattern is 100x100 μm.

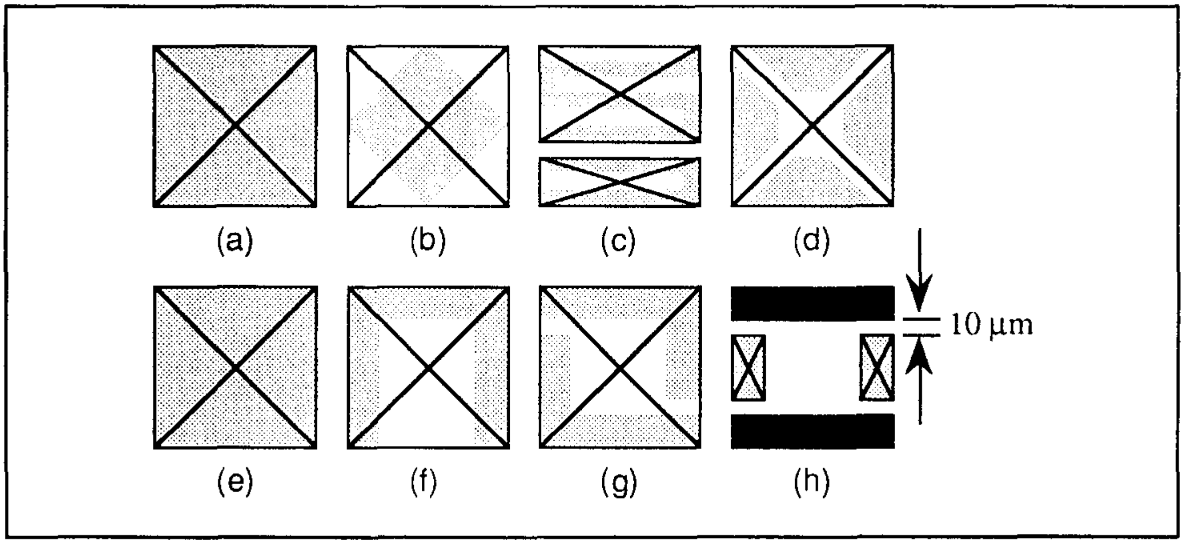

Figure 9. Illustration of the final etched pit for each test pattern.

Thomas J. Jenkins

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John H. Comtois

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DIMENSIONLESS FUN WITH FOAM

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DIMENSIONLESS FUN WITH FOAM

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KEY WORDS: bubbles, capillarity, dimensional analysis, filling, foaming, lather, splashing, suds, surface tension.

PREREQUISITE KNOWLEDGE: Force involves a change of momentum (mass times velocity) with time; more simply, force is mass times acceleration. Tension involves forces pulling in opposite directions on a material. Students may recognize surface tension as "capillarity", the property of a fluid when touching a boundary or interface. Bubbles, soap suds, even plastic foams, are strongly influenced by surface tension, which is a relatively weak force. Changing its boundaries, compositions, or temperatures may significantly change the surface tension of the fluid.

OBJECTIVES: To teach the difference between surface tension (force/length) in a liquid vs tensile strength (force/area) in a solid. To consider capillarity's influence on foam formation and duration. To explore the effect of jet-velocity or nozzle-location on a container-filling station or a garden-fountain display.

INTRODUCTION: Materials labs should include experiments with fluids (liquids, gases) as well as with solids (crystals, glasses). Strange new properties (viscosity, capillarity) should augment familiar concepts (cohesion, adhesion). The "impulse-momentum equation" should replace an "energy-balance", if obvious friction-losses are unknown. Observing a "free-vortex" trail in open-channel flow can elucidate turbulence and illustrate boundary effects.

Industrial processes often involve foam, froth, lather, or suds, which are maintained by low "surface-tensions" (capillarity) and dimensioned as "force per unit-length" (Newtons/metre, Pounds/inch). Indeed, in Statics we study bubbles by drawing free-body diagrams; differentiating between spheres (drops) and shells (bubbles); recognizing foam-factors (additives, interfaces, temperatures); and introducing classics (C.V.Boys, Hunter Rouse).

Suds formation is easy to demonstrate: 1) Bottles are half-filled with hard or soft water; add detergents in various amounts; cork and shake the bottles; then measure the head of suds; and try different water temperatures...2) Copious frothing occurs when root-beer is poured over a scoop of ice-cream in a glass-tumbler; measure the head; try various temperatures and pour-rates...3) Into a pan of water, pour a little kerosene or cooking oil, add a grain of detergent, and watch the radial retreat of that thin-film; try other cleaners and solvents; see which works best at various temperatures.

EQUIPMENT & SUPPLIES: 1) Notebook & calculator,,,2) Ruler, with inch and centimetre divisions...3) Set of similar beakers (say 4-6 sizes)...4) Washtub, with water faucets... 5) Extension hose or overhead pipe, to send a downward jet of water into a beaker...6) Quick-closing valve, at end of hose...7) Makeshift items can be substituted; e.g., plastic tub (sink), hose clamp (valve), or pop-bottle bottoms (beakers).

PROCEDURE: For a simple-but-practical experiment, try filling the containers with a vertical stream of water. Any deep sink with a lever-faucet will do; otherwise, rig a hose-and-bucket setup. Use a matched set of transparent containers, with varying diameter (d) and height (h); beakers, jars, or bottles are fine. When water-temperature (t) is steady, at constant velocity (V), run the jet into a container and fill to overflowing; then abruptly shutoff the faucet. With enough velocity to give a head of foam, which rapidly subsides, water cannot fill the beaker completely. Clearly, the falling jet entrains air to make bubbles in the beaker; the faster the jet and longer the drop, the more the bubbles and less filling. From a convenient datum-plane, the inside-bottom of the beaker, measure the final height (H) of water in the beaker. Also, measure the jet's free-fall distance (D) from faucet-outlet to beaker-bottom. Make several trials, confirm reproducible results, and get a reliable average-height (H) for each beaker trial.

Some jars are fuller than others. Perhaps another jar-shape (J) would have better "fillability" in a different size? Experiment next with various jet-distances, velocities, and temperatures. Take pains to maintain "steady-state" conditions; i.e., constant with time (T). Although more trouble, try comparing different fluids: salt-brine, kerosene, glycerine, or detergents in harder and softer water; perhaps a washing-machine with "suds-saver" is handy in the laundry. Obviously, fluids vary in properties; i.e., density (g), viscosity (v), and capillarity (s). Gathering a variety of data is an interesting challenge.

Next comes the analysis. The optimal jar-size (for maximum filling) should be found for each jet-setting (of known height, velocity, temperature). The obvious application is at a container-filling station, where various jet-distances and velocities may be needed for various containers. Here is where the power and magic of "dimensional analysis" can be exploited, to direct further investigating and to promote efficient reporting.

ALTERNATIVES: For a radical change, invert a beaker and create a "splash-bubble". This phenomenon is the basis of those "shimmering fountains" sold at garden-shops. A vertical stream of water, striking a disk, can produce a beautiful,clear hemisphere, which smoothly cascades into the pool. Students expecting to splatter classmates may experience the serendipity of discovery. Just varying the water-velocity and measuring the resultant bubble-diameter (B) makes an effective investigation, especially if jet-lengths vary. Adding detergent, another variable, should reduce surface-tension and prolong bubble-life.

DIMENSIONAL ANALYSIS: 1) With insight into a problem, recognize all significant variables; avoid duplicating factors, which obscure our analysis...2) Select a basic system of units, usually Mass-Length-Time or Force-Length-Time...3) Add dimensionless shape-factor (J), angle (Radian), or temperature (t) only if variable...4) Systematically consider your fluid's Geometry, Motion, and Forces...5) Identify every pertinent variable, and express each in terms of F-L-T; e.g., surface tension is F/L, velocity is L/T, viscosity is FT/A, A is LxL, and height is L...6) Choose 3-variables to represent F-L-T; don't use our dependent variable (H)...7) For example, let surface tension ($s = F/L$) be F; let velocity ($V = L/T$) be T; and let distance (D) be L...8) Asume H is a function only of D,V,s,v; other factors stay constant (J, h/d, g,t)...9) Make dimensionless groups or parameters (Pi-terms) by applying Buckingham or Rayleigh methods to solve simultaneous equations; here, a simple Factor-Label method can generate our Pi-terms (see the Appendix).

Finally, we can exploit such Pi-terms: 1) The dependent variable (H) can be a function of our other variables (independents); hence a dependent Pi-term becomes a function of the remaining terms...2) A concise two-dimensional plot of our dimensionless groups (with several variables) is now feasible...3) Plots help organize measurements, provide direction for new testing, and show the scope of results... 4) Gaps are then filled in the test data; check basic premises...5) No matter if others used different units; dimensionless plots of similar test-results are directly comparable.

APPENDIX: Let H be a function of J,d,h,D,V,s,v; let D,V,s represent L,T,F. Then Pi-1 =(v)(1/s)(V) =(FT/LL)(L/F)(L/T); Pi-2 =(H)(1/D) =(L)(1/L); and P-3 =h/d =constant. So, our tests are plotted as H/D (on y-axis) versus vV/s (on x-axis), for a family of h/d jars (or J-shapes).

REFERENCES:

1) Boys, C.V., "Soap-Bubbles: Colours and Forces", Dover Inc, NY, 1959.
2) Rouse, Hunter, "Elementary Mechanics of Fluids", John Wiley & Sons, NY, 1946.
3) Allman, W.F., "The Fascinating Physics of Fizz", U.S.News & World Report, Nov.20, 1989.
4) Langhaar, H.L., "Dimensional Analysis and Theory of Models", John Wiley & Sons, NY, 1951.
5) Ipsen, D.C., "Units, Dimensions, and Dimensionless Numbers: The Pitfalls", McGraw-Hill, N.Y., 1960.
6) Widener, E.L., "Dimensional Analysis for Undergraduate Labs", Joint Conf.of N.Central/IN/IL Sections, ASEE.

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INTRODUCTION TO NONDESTRUCTIVE TESTING

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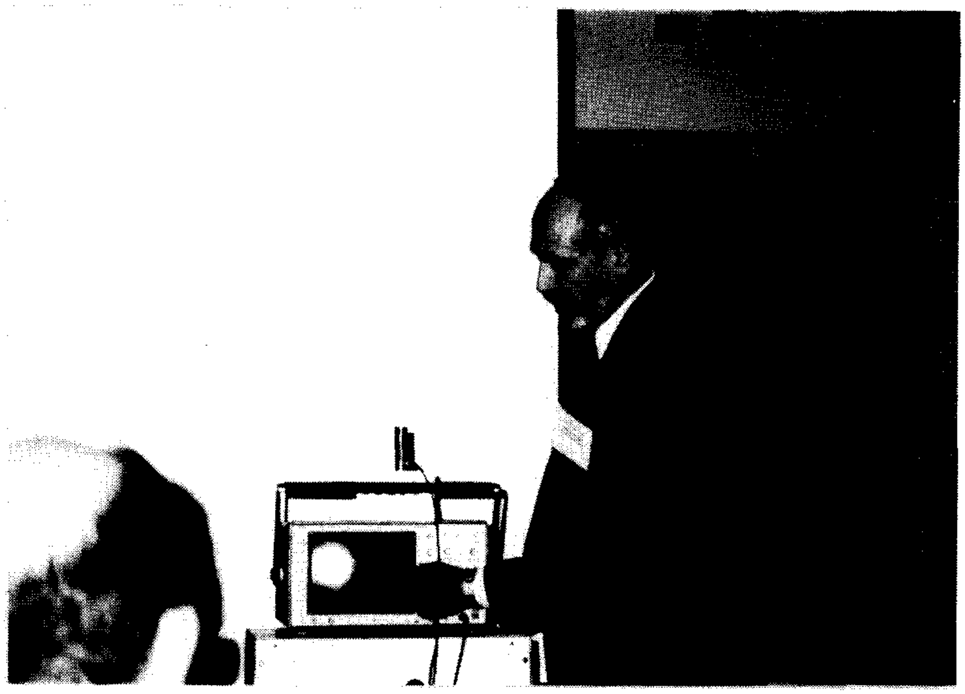

INTRODUCTION TO NONDESTRUCTIVE TESTING

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Key Words: Fracture toughness, critical crack size, ultrasonics, electromagnetics, process flaws.

Prerequisite Knowledge: This experiment should follow a discussion of fracture mechanics, which usually occurs early in an introductory materials course. The student should have prior knowledge of electrical and magnetic theory from a physics course. If more specific discussion of electrical and magnetic properties and failure analysis is presented later in this course or in a second course, this experiment should be delayed until that time.

Objective: To utilize standard test methods for finding flaws in materials and determine the chance that these flaws will result in catastrophic failure.

Equipment:

1. Ultrasonic instrument preset for frequency and material thickness,
2. Ultrasonic transducer, typically 5 MHz and .5" diameter,
3. Couplant (30 wt oil, clear grease, or water-based gell),
4. Aluminum billet stock with artificial flaws and a reference hole in one end,
5. Test bench with power supply for magnetizing a part,
6. Magnetic particles,
7. Steel part with reference notches of known depth and with unkown defects,
8. Conductivity meter with ID probe,
9. Set of standard aluminum, brass, and stainless steel nuts with the same ID as the probe and with varying diameters and hardness,
10. Box of aluminum nuts of the same type as the standards, some with flaws.

Introduction: All materials contain some flaws. We need to know the maximum size flaws that a material can tolerate for the intended stresses that it will experience in service, in order to prevent fatigue or brittle catastrophic failure. We now have a property called fracture toughness that is usefull in this regard. Test methods to accurately measure flaw size are a critical part of this analysis.

Ultrasonic testing (UT) sends a high frequency (1 to 25 MHz) sound pulse into a material and waits for it to return in the pulse-echo mode. Time is used as a measure of the distance that the sound travels. A reflection from the opposite side of a part takes longer to return than the reflection from an internal defect. An oscilloscope which displays time (distance) horizontally and sound intensity vertically will display a spike on the left where some sound reflects from the front surface, a spike on the right from the back surface reflections, and spikes from defects in between.

Magnetic particle testing (MT) can only be used with ferromagnetic materials, primarily steels, because it requires that most magnetic flux lines are contained within a part except where a defect is present near the surface. Flux lines that are forced outward near the defect then can attract magnetic particles that are lightly blown onto the surface. These particles are dyed bright colors so that concentrations of them are easily seen. The size of the defect is determined by the size of the pile of particles and the amperage used to magnetize the part.

Eddy current testing (ET) also is usefull for finding surface defects and depends upon the electromagnetic properties of the material being tested. An alternating current flowing in a small coil will be disturbed by interactions with the material being tested. As the coil is moved along the surface of a part, the disturbance should remain constant. A change indicates a possible flaw or alternatively a change in the composition, microstructure, properties, or surface condition of the material. (For more details, see Askeland or ASM Handbook Vol. 17.)

Procedure:

Ultrasonic Testing

1. Switch on the UT instrument. Note the location of the coarse and fine gain switches, which are the only settings that need to be changed.
2. Apply couplant to the top of the aluminum billet stock, and place the transducer on the billet near the reference hole. Two spikes should appear on the screen. Note that the distance between the spikes on the screen scale is proportional to the thickness of the block.
3. Move the transducer over the reference hole and adjust the gain so that its spike peaks at about mid-height on the screen. This establishes a calibration level for size of defect based upon the reflecting area. Note that hand pressure has a large effect.
4. Increase the gain for scanning and slowly sweep back and forth until you have covered the entire top of the block. Mark the location of suspected defects.
5. Return the gain to the calibration level and estimate the size of defects as a ratio of the reference level. Make a map of the location and size of defects. Compare with a radiograph provided by the instructor. Which of these defects might cause brittle failure if it were on or near the surface of a part machined from this block?

Magnetic Particle Testing

1. Switch on the ultraviolet lamp to let it warm up for 2 minutes. *Warning:* this lamp can get very hot -- you can burn yourself! Turn it off when you are finished.
2. Lock the test part into the test bench, being sure that there is good electrical contact on the copper pads. *Warning:* poor contact may result in arcing.
3. Switch the power supply on and note the amperage. Spray particles onto the top surface for three seconds, then switch the power off. With the black light, look

for and record defects. With your finger wipe the defect indications and note if they reform.

4. Turn the part over and repeat the inspection. Which defects are likely to cause brittle failure?

<u>Eddy Current Testing</u>

1. Calibrate the conductivity meter and probe with standard aluminum nuts that represent the allowable range of inside diameter and hardness. Note the range of conductivity that is considered to be normal. Compare this with nuts made of brass and stainless steel.
2. Check the rest of the aluminum nuts that are provided. If any are out of the calibration range, how might you determine the type of defect that is present?

Notes to the Instructor: To many faculty this experiment may appear to be too mundane and non-scientific, but it has some hidden attributes. First, the objective of finding flaws and considering their potential danger needs to be strongly reinforced. My 20 years of experience in failure analysis suggests that too many engineers assume that safety factors account for all material flaws or even that flaws are not relevant to design. Second, it is a good example of utilizing physical properties for unusual applications. We need to broaden students perspective on the relevancy of <u>all</u> properties and avoid inculcating limited paradigms if we want them to be more effective in their professional careers.

There are nine weekly lab projects in my one quarter introductory materials course, which emphasize the relationship between structure, properties, and processes:

1. tensile testing of steel, aluminum, and a polymer;
2. Charpy testing of annealed mild steel keystock;
3. microstructure and processing of a nail;
4. nondestructive testing;
5. compressive strength of various mixtures of concrete;
6. age hardening of 2024 aluminum;
7. designing aluminum, composite, and wood beams;
8. microwave sintering of tungsten carbide machining inserts; and
9. shear strength of adhesives.

These lab projects were selected to meet most of these criteria:

1. emphasize a lecture topic,
2. low cost and utilize existing equipment,
3. complimentary to other experiments,
4. correlate to other courses,
5. demonstrate emerging technologies, and
6. take advantage of common experience.

The equipment may be the biggest problem for many faculty, since most universities do not have it available. I was able to obtain donations from defense contractors that are downsizing. Your local chapter of ASNT (American Society for Nondestructive Testing) would be a good place to start for donations. MT is the cheapest, since a test bench can be fabricated from a woodworker's vise ($100) and a scavenged power supply with an output of at least 500 amps at 12 volts; or a portable yoke magnet can be purchased for $500. The visible powder costs about $4/lb. A black light and fluorescent powder adds about $200 but with an improvement in sensitivity. My test bar is an automotive camshaft that I scrounged from a local auto shop, and an old lawnmower blade is ideal. No defects were present, so I made some craze-cracks on the hardened lobes by quenching. A diamond saw or laser can be used to create reference defects.

UT instruments and a small assortment of transducers cost a minimum of $7,000, but some manufacturers give educational discounts. ET scopes are also in the same price range, but a $2,000 conductivity meter can be used. A few vendors sell cards that turn a computer into a UT or ET instrument. Test blocks for these methods can be made from aluminum billets with defects provided by drilling .10 to .25 in. dia. holes or sawing slots followed by plugging or filling so that they are not visible. Reference holes should be left visible so that students can accurately calibrate the equipment. A radiograph or drawing of the block should be provided after performing the inspection so that students can check and comment on their results in their written report.

There are many other methods of NDT that can be used. I like to use two methods in the experiment: one for surface and one for internal flaws. Radiography is a lot of fun and gives good detail but is too dangerous for younger students. I use it only in an advanced class. Dye penetrant is cheap and easy to use but very messy -- students quickly have dye on everything. And it doesn't utilize interesting properties. Acoustic-emission instruments may be available from research projects, but otherwise are too expensive and specialized to consider.

References:

Askeland, Donald R.: *The Science and Engineering of Materials, 3rd Ed.*, PWS Publ. Co., 1994, pp. 771-783.

Nondestructive Inspection and Quality Control, ASM Handbook Vol. 17, Ninth Ed., ASM, 1989.

Sastri, Sankar: Fluorescent Penetrant Inspection; Magnetic Particle Inspection; Radiographic Inspection; *National Educators' Workshop: Update 88*, NASA Conference Publication 3060, 1990.

PC LASER PRINTER-GENERATED CUBIC STEREOGRAPHIC PROJECTIONS WITH ACCOMPANYING STUDENT EXERCISE

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projections easily and inexpensively; to detail an implementation of this technique; and to provide an example student exercise demonstrating the use of stereographic projections and their manipulation preparatory to solving applied crystallography problems.

EQUIPMENT: (1) an IBM™ personal computer, 8088, using DOS Version 3.0; (2) the BASICA interpreter, Version 1.10 by Microsoft Corporation™; (3) a Hewlett Packard LaserJet Series II™ laser printer, with extended memory; (4) a Wulff net; (5) a protractor.

PROCEDURE:

Introduction

In any crystal structure, planes and directions can be specified in terms of integers known as Miller indices [1]. The Miller indices for a plane, (hkl), can be found by inverting the intercepts, in terms of the dimension of the unit cell, of the plane with the primary axis system, cleared of fractions and common multipliers. Figure 1 illustrates this for the (120) plane in the cubic system. The Miller indices for a direction, [uvw], can be found by translating the direction to the corner of a unit cell and recording the direction, in terms of the dimension of the unit cell, with reference to the primary axes. Figure 1 also illustrates the [120] direction in the cubic system. The subsequent discussion will be limited to cubic crystals where the normal to the (abc) plane is given by the [abc] direction.

A stereographic projection [2,3] is a two-dimensional representation of the three-dimensional relationship between planes and directions for a given crystal system. The geometric relationship between the planes in the cubic system can be visualized in terms of the actual planes or the normals to the planes. The normal to a plane is referred to as a plane pole (Figure 2). The angle between plane 1 and plane 2, angle θ_1, is numerically equal to the angle between the respective plane poles, angle θ_2. The intersection of each plane with a spherical surface generates a great circle. A great circle has a diameter equal to the sphere diameter. The intersection of the companion plane pole with the spherical surface generates a point which is normal to the plane-great circle. This can be measured on another great circle containing the plane pole-point and a point on the corresponding plane-great circle as shown in Figure 3. The projection of the plane-great circles and

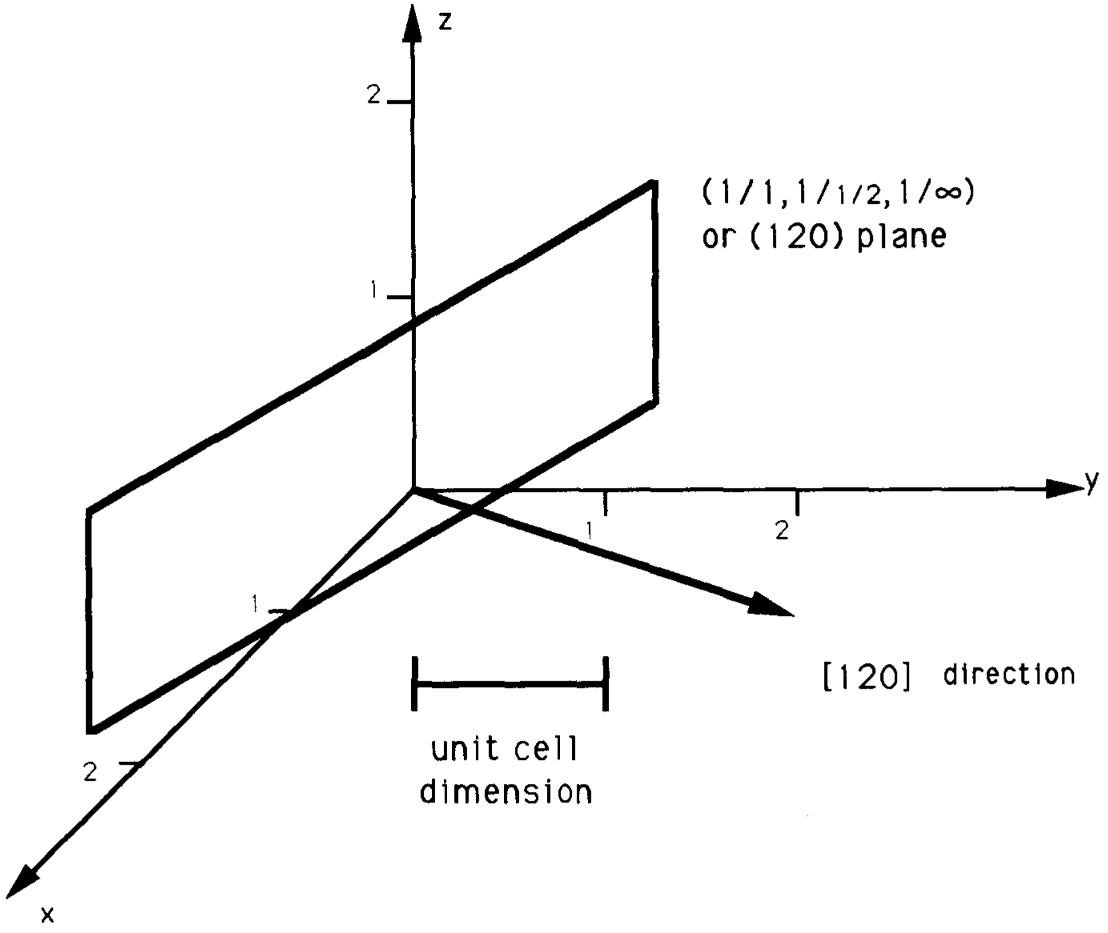

Figure 1. Miller indices example for the cubic system.

plane pole-points from the surface of the sphere to a plane surface creates a stereographic projection [4,5].

The plane pole at the center of a stereographic projection has its corresponding plane-great circle as the exterior circle which is the outside boundary of the projection. The assignment of a second direction will fix the orientation of the particular projection. Figure 4 illustrates this for a central [001] pole and two other poles which form a right-handed coordinate system. The corresponding great circle for each pole is also noted. The angle between any two planes can be determined from a stereographic projection containing both plane poles by positioning both plane poles on the same great circle, superimposing the circle on a Wulff net [6,7], to measure the angle. Figure 4 also contains the $[\bar{1}01]$ plane pole and corresponding great circle. The angle between the [101] plane pole and the [111] plane pole can be determined from the $(\bar{1}01)$ great circle using the Wulff net or alternatively the equation below. The angle between the $(h_1k_1l_1)$ and $(h_2k_2l_2)$ planes, which is equivalent to the angle between the $[u_1v_1w_1]$ and $[u_2v_2w_2]$ directions, is given by

$$\text{angle} = \cos^{-1}\left(\left(h_1 * h_2 + k_1 * k_2 + l_1 * l_2\right) \Big/ \left(\left(h_1^2 + k_1^2 + l_1^2\right)\left(h_2^2 + k_2^2 + l_2^2\right)\right)^{1/2} \right). \quad (1)$$

<u>Theory</u>

The theoretical basis for the computer generation of stereographic projections has been detailed by Johari and Buchanan [8], Johari and Thomas [9], Kosel [10,11], and Staley [12,13]. Given a particular [hkl] plane pole, the location of this pole on a particular stereographic projection can be determined by first computing the cosine of the angle, between the [hkl] direction and each axis of the right-handed coordinate system of the desired stereographic projection. The central plane pole for this technique is designated as [abc], while the other plane poles which define the orientation are designated as [def] and [uvw]. The vector cross product of the second set must generate the central plane pole (i.e., [def] x [uvw] = [abc]). Equation (2), which is configured for the central pole, [abc], of the desired projection, is used to compute the cosine of the angle between [hkl] and [abc], designated as α, following the notation of Kosel [10].

$$\cos(\alpha) = \left(h * a + k * b + l * c\right) \Big/ \left(\left(h^2 + k^2 + l^2\right)\left(a^2 + b^2 + c^2\right)\right)^{1/2} \quad (2).$$

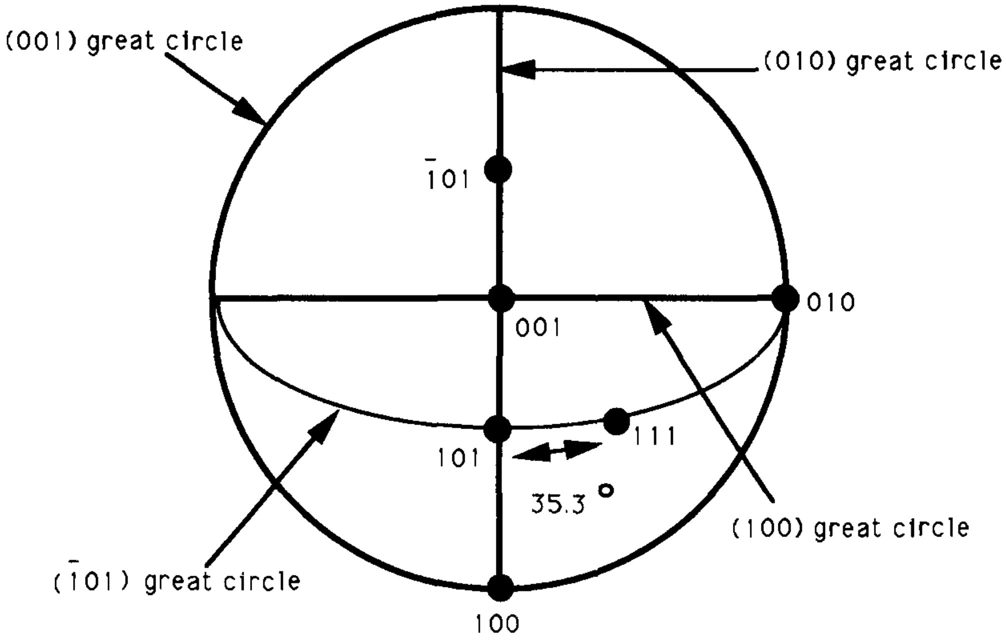

Figure 4. (001) cubic stereographic projection

The other two angles are designated [10] as δ, relative to the [def] direction, and μ, relative to the [uvw] direction. Given these angles, the location of any plane pole on the particular projection can be computed using the equations below which generate an x-y location on the given stereographic projection relative to an origin in laser printer coordinates (XP,YP), where R is the radius of the projection in laser printer coordinates and AR is the aspect ratio for locating positions along the y-axis:

$$\text{Xlocation} = R*\cos(\mu)/(1.0 + \cos(\alpha)), \text{ and} \tag{3}$$

$$\text{Ylocation} = R*AR*\cos(\delta)/(1.0 + \cos(\alpha)). \tag{4}$$

Figure 5 illustrates the stereographic projection with the following indicated: (1) x-y directions; (2) coordinate system origin in laser printer coordinates (0,0); and (3) stereographic projection origin (XP,YP). Any plane pole which generates an x-y location which would fall outside the great circle belonging to the central plane pole, which is the boundary of the projection, corresponds to a plane pole whose location would be on the back of the sphere and thus would not appear on the desired projection. A great circle corresponding to a given plane pole can then be drawn by locating points on the circle and displaying only those points which would appear on the projection.

Plot Generation

The original Basic program was developed on an IBM™ PC/XT computer with DOS Version 3.10 and using the accompanying Version 1.10 by Microsoft Corporation™ BASICA.COM Basic language interpreter. The Hewlett Packard LaserJet Series II™ laser printer with 2.56 Mbytes of internal memory, connected to the computer using a parallel interface, and configured to factory default settings was used to generate hard copies of the stereographic projection (Instructor Note 2). The program appears in Appendix A.

The program is loaded and started using the Basic language interpreter. The user must enter the central plane pole first, [abc] in the program, with a comma between each digit. Two other plane poles, [def] and [uvw] in the program, must also be entered with a comma between each digit. Next, the user is given the option of displaying the pole indexes, with a bar over a number indicating a negative number. The user is next asked to enter a file name for the ASCII file which contains a list of the plane poles to be

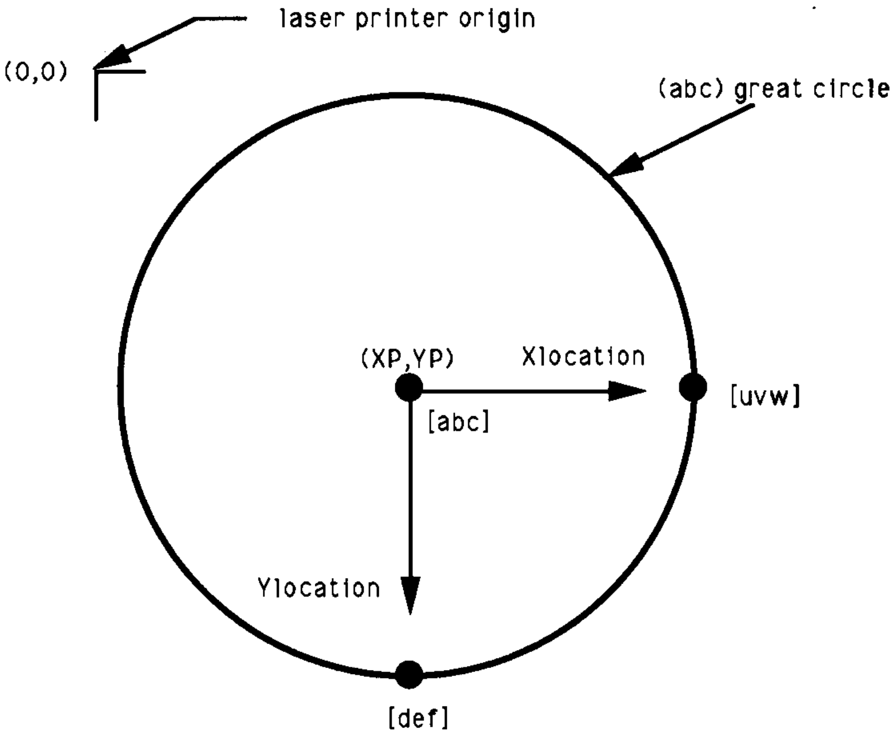

Figure 5. [abc] stereographic projection with coordinate system identified.

included in the plot. This file is created beforehand using one of the text editors which accompanies DOS, EDIT.COM or EDLIN.COM. The file must contain one plane pole per line with a comma separating the numbers, a negative index should be preceded by a minus sign. An additional digit, preceded by a comma, must be included to specify whether the corresponding great circle will be included in the plot. If this last number is a "0" the great circle will not be plotted. While other entries are possible, the program suggests that a "1" be included when the drawing of a great circle is desired. The last line of this file must be four zeros separated by commas. Any poles which are included in this file that would not correctly appear on the projection that was requested are ignored by the program. Appendix B contains an example ASCII file.

EXAMPLE STUDENT EXERCISE (Instructor Note 3):

After a review of classroom material on Miller indices, stereographic projections, and the use of a Wulff net, the following exercises are assigned:

(1) generate one of three standard projections (100), (010), or (001); this requires the selection of the other two axes such that the vector cross product generates the central pole with the use of ASCII file hklall1, which contains all possible plane poles containing -1, 0, or 1 (Instructor Note 4) .

(2) determine the angle between two specific plane poles using both Equation (1) and a Wulff net.

(3) sketch both great circles belonging to these poles and using a protractor to measure the angle between the tangents of these great circles at the point of intersection which is equal to the angle computed using Equation (1) and the angle measured with the Wulff net.

(4) start with a (001) projection with only (hkl) that are -1,0, or 1 and then establish the rotations undergone by selected poles to create a (011) projection with similar hkl's and then obtain it.

(5) determine the rotation of the above (011) projection that would be required to generate a (111) projection and then obtain it.

SAMPLE PLOTS AND RESULTS:

The (001) standard projection, where [def] = [100] and [uvw] = [010], appears in Figure 6. The great circles corresponding to the (011) and (111) plane poles are shown. The angle between these planes can be determined to be 35.3° using Equation (1) or the Wulff net using the great circle corresponding to the $(0\bar{1}1)$ plane pole. The corresponding great circle tangents are also sketched with the angle measurement indicated. Additionally, the rotations necessary for three selected poles to create a (011) projection are indicated. A resulting (011) standard projection, where [def] = [100] and [uvw] = $[01\bar{1}]$, is given in Figure 7. Included are the rotations necessary to create a (111) standard projection for three selected poles. Figure 8 shows this new projection, where [def] = $[2\bar{1}\bar{1}]$ and [uvw] = $[01\bar{1}]$.

INSTRUCTOR NOTES:

(1) Although no longer covered in most introductory materials science textbooks, stereographic projections are a powerful tool to examine geometric relationships between crystal planes and directions. It is useful for sophomore students to become acquainted with stereographic projections in a laboratory exercise in order to become more familiar with the crystallographic topics covered in one or more accompanying lectures. Generally, students grasp fundamental aspects but often lack the confidence to solve applied problems in crystallography. However, this is remedied when students encounter the use of stereographic projections in subsequent upper-level materials science courses (e.g., companion paper in this Workshop [14].

(2) The only major consideration with this procedure is that the density of plotting is limited by the amount of random access memory in the printer. This can be overcome by increasing memory, but there is some upper limit dictated by individual machines. The original code was developed for an early-model PC and laser printer. In lieu of tracing the technology of the PC and the evolution of laser printers, a current-day PC and printer were tested for compatibility. An IBM PS/Value Point 425/SX PC running DOS 5.0 and Windows 3.1 was used for testing. The DOS directory contains a Basic interpreter IBM^{TM} Basic Ver. a3.4 which is the file BASICA.COM. In addition, a Hewlett Packard LaserJet IIIP^{TM}, using Printer Control Language 5 (PCL5), was used to generate hard copies. Following the same procedures detailed in the section on Plot Generation will

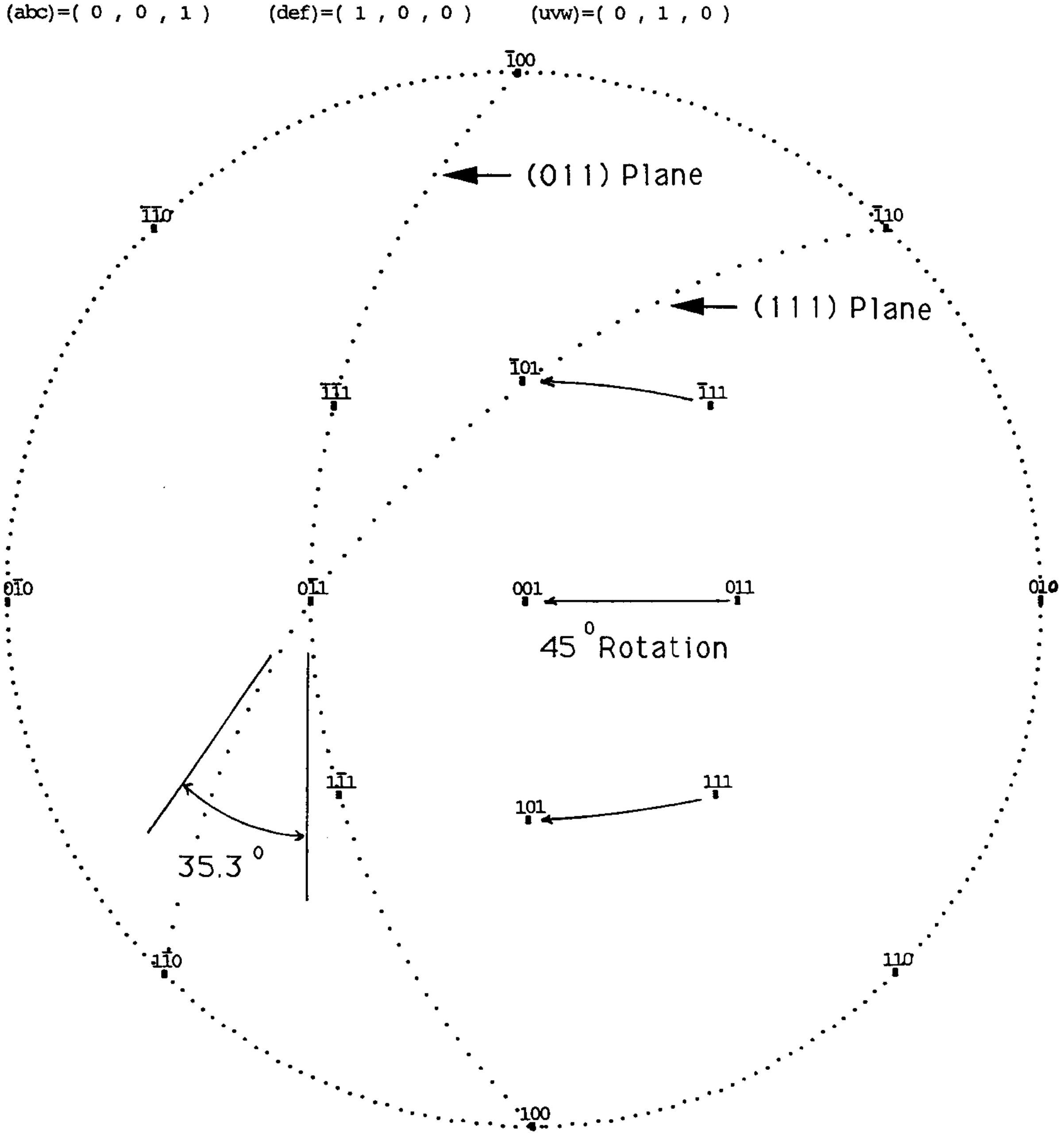

Figure 6. (001) cubic standard projection.

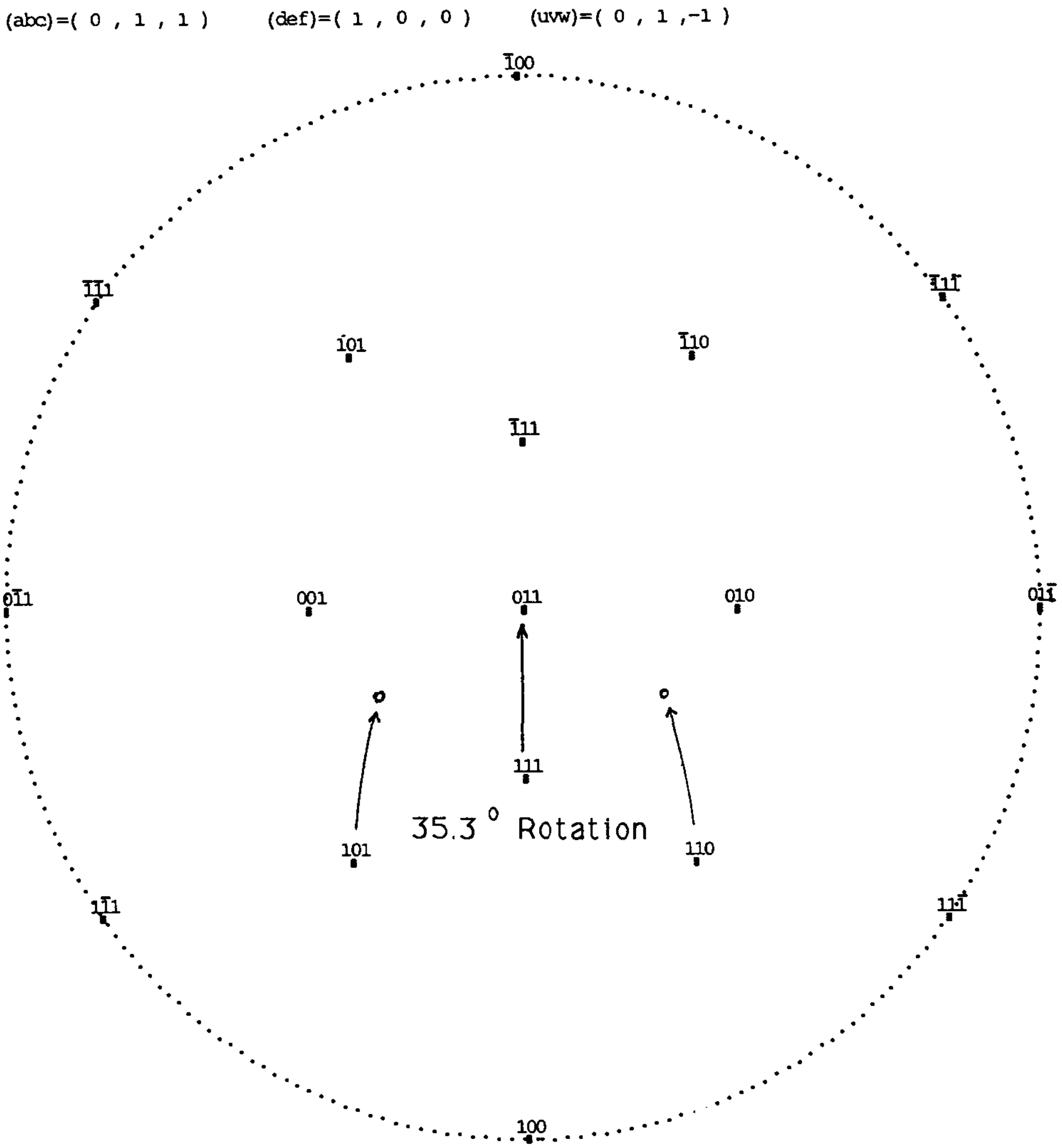

Figure 7. (011) cubic standard projection.

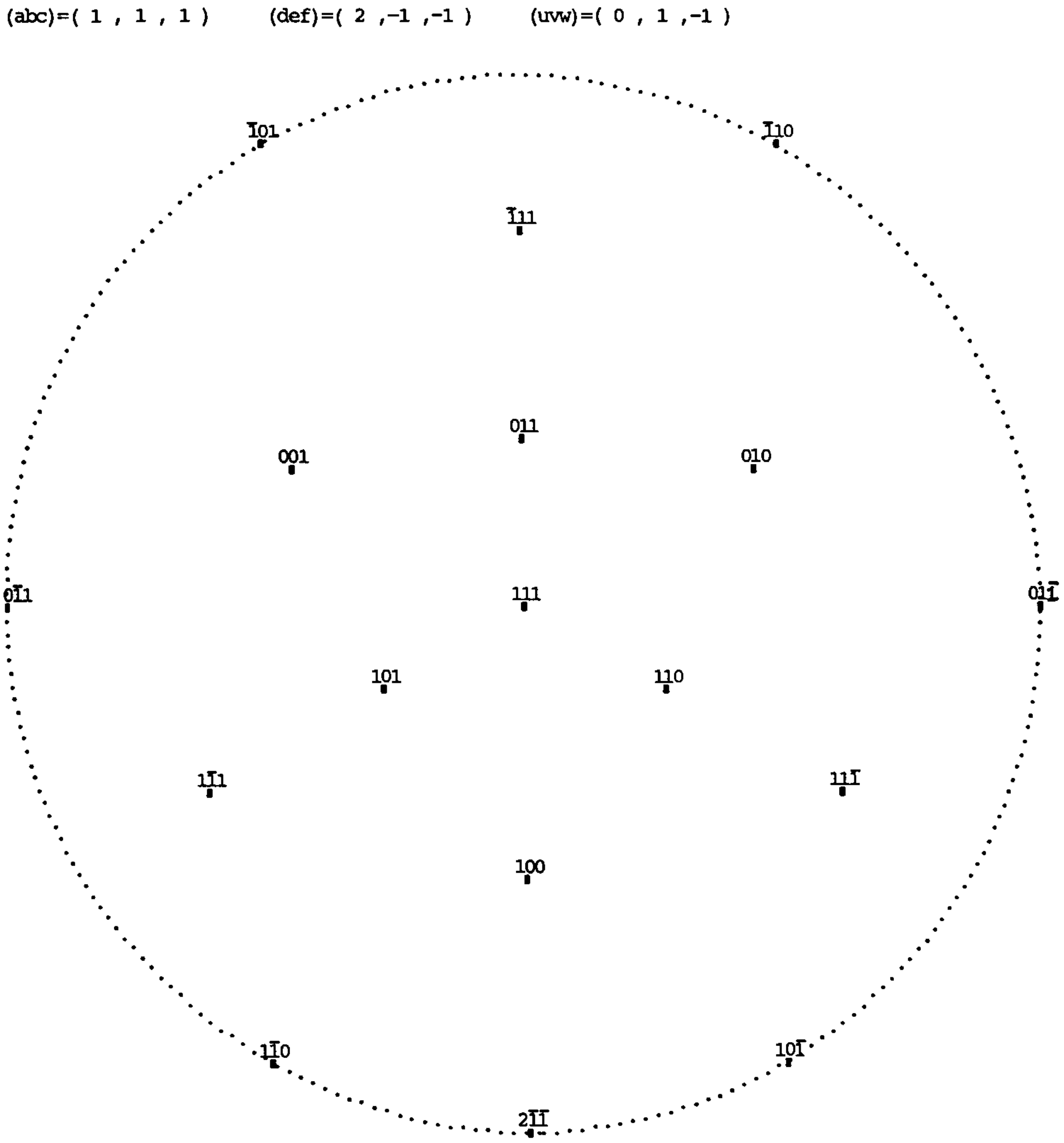

Figure 8. (1 1 1) cubic standard projection.

create the same stereographic projections, but there is still a memory limitation. The key feature for portability is the laser printer. Any printer which supports PCL5 should generate the same projections given that the connected PC can generate the same sequence of characters as the program in Appendix A. The original code was developed before PCL5 was established, but upward compatibility was maintained by Hewlett Packard as its laser printer family developed.

(3) A number of additional crystallography problems with solutions are provided by Moon [15].

(4) The (100), (010), and (001) projections are generated using the program in Appendix A and the input ASCII file, referred to as hklall1, in Appendix B. The program in Appendix A can be easily adapted to the tetragonal and orthorhombic crystal systems. This will require the generation of separate plane pole and direction plots.

REFERENCES:

[1] Van Vlack, L. H.: Elements of Materials Science and Engineering, Fifth Edition. Addison-Wesley Publishing Company, Inc., 1985, pp. 84-90.

[2] Cullity, B. D.: Elements of X-ray Diffraction, Second Edition. Addison-Wesley Publishing Company, Inc., 1987, pp. 63-78.

[3] Barrett, C.; and Massalski, T. B.: Structure of Metals Crystallographic Methods, Principles and Data, Third Revised Edition. Pergamon Press, 1980, pp. 30-43.

[4] Cullity, B. D.: op. cit., p. 65.

[5] Barrett, C.; and Massalski, T. B.: op. cit., p. 32.

[6] Cullity, B. D.: op. cit., p. 67.

[7] Barrett, C.; and Massalski, T. B.: op. cit., p. 35.

[8] Johari, O.; and Buchanan, W.: Computer Program for Obtaining Standard Projections of Planes and Directions in Any Crystal System. Trans. Met. Soc. AIME, vol. 236, no. 12, 1966, pp. 1760-1761.

[9] Johari, O.; and Thomas, G.: The Stereographic Projection and Its Applications. Techniques of Metals Research, Editor: Bunshah, R. F., Volume IIA, Interscience Publishers, 1969.

[10] Kosel, T. H.,: Computational Techniques for Stereographic Projection. J. Mater. Sci., vol. 19, no. 12, 1984, pp. 4106-4118.

[11] Kosel, T. H. , : Microcomputer Stereographic Projection. J. Metals, vol. 37, no. 3, 1985, pp. 56-57.

[12] Staley, J. T.: PC Program for Materials Science - Part II. J. Metals, vol. 37, no. 3, 1985, p. 71.

[13] Staley, J. T.: PC Program for Materials Science - Part III. J. Metals, vol. 37, no. 4, 1985, p. 76.

[14] Elban, W. L.: Stereographic Projection Analysis of Fracture Plane Traces in Polished Silicon Wafers for Integrated Circuits. National Educators' Workshop: Update 94, NASA CP- . 1994.

[15] Moon, J. R.: Worked Examples in Stereographic Projection. Monograph No. 2, The Institution of Metallurgists, 1978.

SOURCES OF SUPPLIES (1994 Prices): The Wulff net, made of plastic, is available from Polycrystal Book Service, P. O. Box 3439, Dayton, OH 45401-3439 at a cost of $20.00. A few (i.e., (001), (111), (211), (011), and (130)) standard cubic projections (18 cm diameter) can also be purchased from Polycrystal for $15.00 each.

ACKNOWLEDGMENT: The Office of Naval Research under Work Request Number N00014-87-K-0175 originally funded software development to create stereographic projections for orthorhombic ammonium perchlorate crystals. Subsequently, the software was modified to create cubic projections for instructional purposes.

APPENDIX A:

Program CUBICSP . BAS

```
1 REM          STEREOGRAPHIC PROJECTION
10 REM          CUBIC CRYSTAL LASER OUTPUT
20 X=1!
30 Y=1!
40 Z=1!
45 PRINT "PUT LASER PRINTER ON LINE "
50 PRINT "ENTER THREE PERPENDICULAR PLANES WHOSE NORMALS SATISFY "
60 PRINT "       [def] X [uvw] = [abc]"
70 INPUT "(abc)";A,B,C
80 INPUT "(def)";D,E,F
90 INPUT "(uvw)";U,V,W
92 PRINT "DO YOU WISH TO DISPLAY POLE INDEXES (Y=1 N=0) "; : INPUT ANS
94 PRINT "INPUT (hkl) FILE NAME => "; : INPUT F$
95 REM SET LASER PRINTER FOR LETTER SIZE,NO TOP MARGIN,AND H+V SPACING
96 PRINT "HOW MANY COPIES ? "; : INPUT ICOUNT
97 FOR I=1 TO ICOUNT
98 OPEN "I",#1,F$
100 LPRINT CHR$(27)+"&l2a0e1C": LPRINT CHR$(27)+"&k1H"
105 R=475 'radius of circle
106 TH=6.3 'angle,in radians, for complete circle draw
107 AR=.4 'aspect ratio for Laser Printer
108 DELTH=250! 'default TH increment factor
110 XLOC=475: YLOC=275: RAD=475: GOSUB 1000 'draw major circle
115 GOSUB 5000 ' locate three major poles on circle
120 X2=X*X : Y2=Y*Y : Z2=Z*Z
130 C1=SQR( A*A/X2 + B*B/Y2 + C*C/Z2 )
140 C2=SQR( U*U/X2 + V*V/Y2 + W*W/Z2 )
150 C3=SQR( D*D/X2 + E*E/Y2 + F*F/Z2 )
160 INPUT #1,H,K,L,DGC
162 IF (H=0) THEN IF (K=0) THEN IF (L=0) THEN GOTO 9000 'end of input file
170 H2=H*H : HA=H*A : HU=H*U : HD=H*D
190 K2=K*K : KB=K*B : KV=K*V : KE=K*E
210 L2=L*L : LC=L*C : LW=L*W : LF=L*F
215 C4=SQR( H2/X2 + K2/Y2 + L2/Z2 )
220 COAL=( HA/X2 + KB/Y2 + LC/Z2 )/( C1*C4 )
230 COMU=( HU/X2 + KV/Y2 + LW/Z2 )/( C2*C4 )
240 CODE=( HD/X2 + KE/Y2 + LF/Z2 )/( C3*C4 )
242 AAL=ABS(COAL)
245 IF (AAL<1.00001 AND AAL>.99999) GOTO 330 ' (abc) and compliment are ignored
250 XP=475+INT((R*COMU/(1!+COAL))+.5)
260 YP=275+INT((R*AR*CODE/(1!+COAL))+.5)
264 AXP=XP-475
265 AYP=(YP-275)/AR : RAXY=INT(SQR(AXP*AXP+AYP*AYP)+.5)
266 IF( RAXY> 476 ) GOTO 330 ' pole on back projection, ignore .
267 XLOC=XP: YLOC=YP:  GOSUB 4000 ' plot pole
269 IF(DGC=0) GOTO 330 ' do not draw great circle
270 IF(COAL=0)  THEN GOSUB 3000 ' projection of circle is a straight line
271 IF (COAL=0) GOTO 330
```

```
272 XP=XP-475 : YP=(YP-275)*(1!/AR) : RGC=INT(R/ABS(COAL)+.5)
273 DOC=R*ABS(SQR(1-COAL*COAL)/COAL) : IF(RGC>1500) THEN DELTH=1000!
274 XC=475+INT(DOC*XP/SQR(XP*XP+YP*YP)+.5)
275 YC=275+INT(AR*DOC*YP/SQR(XP*XP+YP*YP)+.5)
276 XLOC=XC: YLOC=YC: RAD=RGC: GOSUB 2000 ' draw great circle
280 DELTH=250!
330 GOTO 160
400 LPRINT CHR$(27)+"&l0H"
405 CLOSE #1
410 NEXT I
420 GOTO 9999
1000 REM       CIRCLE DRAW SUBROUTINE
1010 DTH=0!
1020 C$=STR$(INT(XLOC+RAD*1!*SIN(DTH)+.5))
1030 R$=STR$(INT(YLOC+RAD*AR*COS(DTH)+.5))
1040 LPRINT CHR$(27)+"&a"+R$+"r"+C$+"C";: LPRINT "."
1050 DTH=DTH+TH/250!
1060 IF(DTH<=TH) GOTO 1020
1100 RETURN
2000 REM          ARC DRAW SUBROUTINE
2010 DTH=0!
2020 CN=INT(XLOC+RAD*1!*SIN(DTH)+.5)
2025  CNC=CN-475
2030 RN=INT(YLOC+RAD*AR*COS(DTH)+.5)
2035  RNR=(RN-275)/AR
2040  IF(SQR(CNC*CNC+RNR*RNR) > 475) GOTO 2080
2050  C$=STR$(CN)
2060  R$=STR$(RN)
2070  LPRINT CHR$(27)+"&a"+R$+"r"+C$+"C";:LPRINT "."
2080 DTH=DTH+TH/DELTH
2090 IF(DTH<=TH) GOTO 2020
2999 RETURN
3000 REM          LINE DRAW SUBROUTINE
3005 P1=475+AYP : Q1=275-AXP*AR : P2=475-AYP : Q2=275+AXP*AR
3010 DP=(P2-P1)/100! : DQ=(Q2-Q1)/100!
3012 FOR I=0 TO 100
3015 P1R=P1-475 : Q1R=(Q1-275)/AR
3016 RPQ=SQR(P1R*P1R+Q1R*Q1R)
3017 IF( RPQ > 475 ) GOTO 3040
3020 C$=STR$(INT(P1)) : R$=STR$(INT(Q1))
3030 LPRINT CHR$(27)+"&a"+R$+"r"+C$+"C"; : LPRINT "."
3040 P1=P1+DP : Q1=Q1+DQ
3100 NEXT I
3500 RETURN
4000 REM          POINT DRAW SUBROUTINE
4010 FOR IX=XLOC-1 TO XLOC+1
4020 FOR IY=YLOC-1 TO YLOC+1
4030 C$=STR$(INT(IX)) : R$=STR$(INT(IY))
4040 LPRINT CHR$(27)+"&a"+R$+"r"+C$+"C"; : LPRINT "."
4050 NEXT IY
4060 NEXT IX
4070 IF(ANS=0) GOTO 4500
4200 C$=STR$(INT(XLOC-8)): R$=STR$(INT(YLOC-3))
4210 LPRINT CHR$(27)+"&a"+R$+"r"+C$+"C";
```

```
4220 LPRINT STR$(ABS(H))+SPACE$(7)+STR$(ABS(K))+SPACE$(7)+STR$(ABS(L))
4230 C$=STR$(INT(XLOC-8)) : R$=STR$(INT(YLOC-6))
4240 H$=" " : K$=" " : L$=" "
4250 IF(H<0) THEN H$="-"
4260 IF(K<0) THEN K$="-"
4270 IF(L<0) THEN L$="-"
4280 LPRINT CHR$(27)+"&a"+R$+"r"+C$+"C";
4290 LPRINT H$+SPACE$(9)+K$+SPACE$(9)+L$
4500 RETURN
5000 REM          AXIS POINTS DRAW SUBROUTINE
5010 H=A: K=B: L=C: GOSUB 4000
5020 XLOC=950: YLOC=275
5030 H=U: K=V: L=W: GOSUB 4000
5040 XLOC=475: YLOC=465
5050 H=D: K=E: L=F: GOSUB 4000
5500 RETURN
9000 REM          EXIT AND "ID" CODE
9010 LPRINT CHR$(27)+"&l2D" : LPRINT CHR$(27)+"&k10H"
9020 LPRINT CHR$(27)+"&a1r1C";
9025 LPRINT "CUBIC PROJECTION ON ";DATE$;" AT ";TIME$;"  using file ";F$
9030 LPRINT CHR$(27)+"&a2r1C";
9040 LPRINT "(abc)=(";A;",";B;",";C;")";:
9050 LPRINT "     (def)=(";D;",";E;",";F;")";:
9060 LPRINT "     (uvw)=(";U;",";V;",";W;")";: 9070 GOTO 400
9999 END
```

APPENDIX B:

ASCII File hklall1

```
-1 ,-1 ,-1 , 0
-1 ,-1 , 0 , 0
-1 ,-1 , 1 , 0
-1 , 0 ,-1 , 0
-1 , 0 , 0 , 0
-1 , 0 , 1 , 0
-1 , 1 ,-1 , 0
-1 , 1 , 0 , 0
-1 , 1 , 1 , 0
 0 ,-1 ,-1 , 0
 0 ,-1 , 0 , 0
 0 ,-1 , 1 , 0
 0 , 0 ,-1 , 0
 0 , 0 , 1 , 0
 0 , 1 ,-1 , 0
 0 , 1 , 0 , 0
 0 , 1 , 1 , 0
 1 ,-1 ,-1 , 0
 1 ,-1 , 0 , 0
 1 ,-1 , 1 , 0
 1 , 0 ,-1 , 0
 1 , 0 , 0 , 0
 1 , 0 , 1 , 0
 1 , 1 ,-1 , 0
 1 , 1 , 0 , 0
 1 , 1 , 1 , 0
0,0,0,0
```

STEREOGRAPHIC PROJECTION ANALYSIS OF FRACTURE PLANE TRACES IN POLISHED SILICON WAFERS FOR INTEGRATED CIRCUITS

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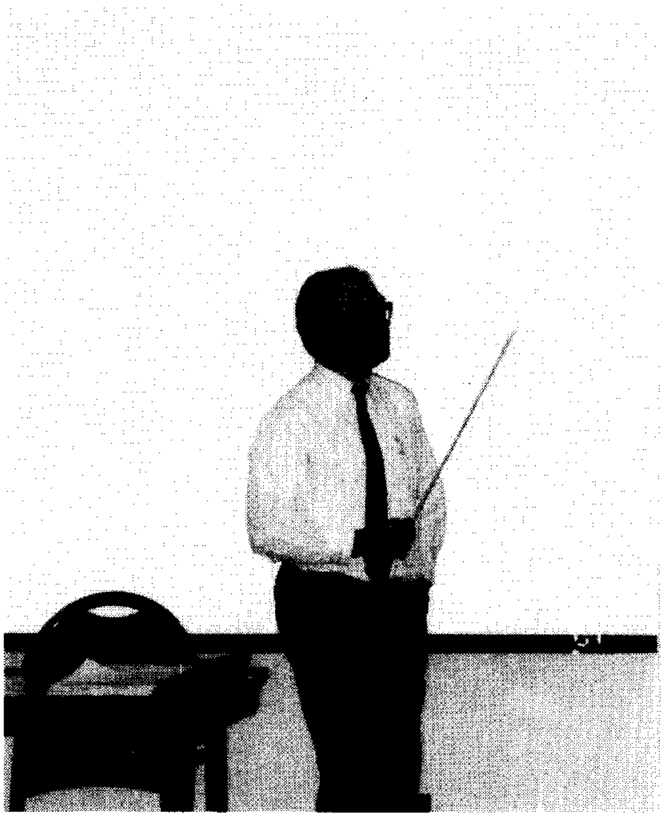

STEREOGRAPHIC PROJECTION ANALYSIS OF FRACTURE PLANE TRACES IN POLISHED SILICON WAFERS FOR INTEGRATED CIRCUITS

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ABSTRACT: A procedure is described for crystallographically assessing probable fracture planes observed in polished wafers used in integrated circuit manufacturing. The wafers had been sliced from a silicon single crystal grown by the Czochralski method. To confirm the wafer orientation as provided by the manufacturer, a back-reflection Laue x-ray diffraction photograph was obtained, and a zone analysis was performed. The wafers are reasonably fragile and can be broken into large fragments, making the intersection of the fracture planes with the polished surface suitable for characterization. A single-trace analysis, involving the use of a standard cubic stereographic projection, was performed on: (1) a simple (crystallographic-appearing or cleavage) fracture for one wafer and (2) a more complex fracture (i.e., partially non-crystallographic-appearing with straight segments) for a second wafer. This allowed the identification of a number of possible fracture planes from which the most likely were selected by comparison with what is reported in the literature for silicon. The cleavage fracture in the first wafer and three straight segments of the complex fracture in the second wafer are consistent with {111} fracture planes. A two-trace analysis on the wafer with the cleavage fracture confirmed this plane.

KEY WORDS: crystallography, single crystal, back-reflection Laue x-ray diffraction, stereographic projection, trace analysis, fracture, cleavage, silicon.

PREREQUISITE KNOWLEDGE: upper-level undergraduate laboratory experiment requiring basic knowledge of crystallography, x-ray diffraction, and fracture behavior as described in an introductory materials science course.

OBJECTIVES:

(a) Experimental Goals:

1. to determine the orientation of a polished wafer of single crystal silicon using the back-reflection Laue x-ray diffraction technique and

2. to perform a stereographic projection analysis of fracture plane traces in silicon wafers to identify probable candidate planes.

(b) Learning Goals:

1. to become familiar with a widely used x-ray diffraction method for orienting single crystals;

2. to become familiar with stereographic projections and their use in applied crystallography problems;

3. to become familiar with the appearance of crystallographic versus non-crystallographic fracture in brittle materials;

4. to become familiar with trace analysis to identify important crystallographic features in single crystals; and

5. to become familiar with the materials science literature in order to extract relevant crystallographic information.

EQUIPMENT AND SUPPLIES: (1) Polished single crystal silicon wafers (100 mm diameter x 0.54 mm thick); (2) Standard x-ray generator with back-reflection Laue apparatus providing diffraction photograph; (3) Tracing paper; (4) Greninger chart; (5) Wulff net; (6) Standard cubic stereographic projections -- (001), (110), and (111).

PROCEDURE:

Introduction

Single-crystal materials are increasingly being adopted in technological applications. Examples include turbine blades in jet engines and semiconducting materials in a variety of electronic devices. The ability to orient single crystals and to identify their crystallographic features are important skills to acquire. The determination of the orientation of a single crystal is often accomplished using the Laue x-ray diffraction method in either back-reflection or through-transmission arrangement [1,2]. To analyze the resultant diffraction pattern, a two-dimensional circular plot of three-dimensional angular relationships for crystal directions and planes, known as a stereographic projection [3,4], is used (Instructor Note 1). In this plot, directions and plane poles are represented by points while planes appear as great circles. Stereographic projections are also useful in characterizing crystallographic features such as growth facets, slip bands, growth and deformation twins, cracks, and etch pit arrays. Relevant planes/directions for such features can be identified by analyzing their traces (i.e., the intersection of the plane associated with a particular feature with the as-viewed surface).

For single crystals of brittle materials, fracture often occurs along specific crystallographic planes, termed cleavage planes. Lawn and Wilshaw [5] provide a list of the primary cleavage planes for a number of materials. Cleavage fracture is identified by the appearance of straight, crystallographic traces. Fracture in brittle crystals can also be non-crystallographic or amorphous in character with traces that are curved or highly irregular.

Large ($\geq$ 76 mm diameter) polished single-crystal silicon wafers [6] are used in considerable quantity in integrated circuit (IC) manufacturing. The wafers are sliced from an ingot grown by the Czochralski method. A crystallographic reference flat (Fig 6.1, Mayer and Lau [7]) is ground on the ingot prior to slicing. Because of their thinness and the brittleness of silicon, the wafers are reasonably fragile, and breakage can occur on handling.

This work consists of performing a back-reflection Laue experiment to substantiate the orientation of the wafers as given by the manufacturer. A stereographic projection analysis of fracture plane traces is then performed on wafers that have been broken. The trace analysis provides a number of candidate planes from which the most probable are selected on the basis of what has been reported in the literature. In one instance, the necessary two-trace analysis to confirm the plane was performed (as an option). The following problem statement is given:

> You have recently been hired as a materials scientist by a company that manufactures polished single crystal silicon wafers for use as the IC substrate material. Unfortunately, an unusually large number of wafers have been breaking. As a first step in failure analysis, you have been asked to identify the fracture planes that result. It is of interest to investigate the fracture that resulted in two wafers:
>
> (1) the simple fracture in Wafer #1; and
> (2) the more complex fracture in Wafer #2.

A. BACK-REFLECTION LAUE EXPERIMENT (Instructor Note 2)

1. Mount the entire wafer or a portion containing the reference flat on a goniometer (Instructor Note 3). The orientation of the wafer must be noted (Instructor Note 4).

2. Obtain a Laue photograph (Instructor Note 5).

Data Analysis

1. Study the shape of the diffraction spots recorded on the film [8-9]. Sharp, circular spots indicate good perfection in the small volume of the crystal that was sampled. Streaked spots (i.e., spots with asterism) reveal that the crystal is strained.

2. Since the photograph was taken in back-reflection, it must be read through its back to relate properly to the as-viewed wafer surface whose orientation was previously noted. The film reading can be accomplished by pressing a needle into the center of the spots to be analyzed and then marking each spot on the film back with a black felt tip marker. Transfer the spots to tracing paper by placing it on top of the back of the film while lightly denoting the outline of the film with its top being up.

3. For six (6) prominent zones of spots, measure the angle of inclination, ϕ, and the azimuthal angle, α, using a Greninger chart and following in detail the procedure described by Barrett and Massalski [10].

4. Using a Wulff net and again referring to the procedure in Barrett and Massalski, plot the zone axes on a stereographic projection paper. This can be accomplished by imposing the axes on the same tracing paper containing the previously recorded Laue spots. In this case, care must be taken to ensure that the centers of the Laue photograph and stereographic projection coincide.

5. The crystal surface incident to the x-ray beam is identified by comparing the positions of the zone axes with the locations of directions denoted in the various computer-generated [11] standard cubic stereographic projections provided (i.e., (001), (110), and (111)) -- (Instructor Note 6). Close coincidence for all six (6) axes must occur to establish the identity of the irradiated surface (Instructor Note 7).

B. IDENTIFICATION OF PROBABLE FRACTURE PLANES

1. Trace the outlines for the fragments of the two wafers provided (Instructor Note 8).

2. Examine the perimeter of the wafer fragments. Distinguish those portions that are curved or irregular from straight-line segments. The exterior growth surface of the crystal is curved and non-crystallographic fracture yields curved or irregular traces. The straight-line (i.e., crystallographic) traces result from the intersection of either a fracture plane or the reference flat with the as-viewed polished surface. Referring to the procedure described by Barrett and Massalski [12], extend those crystallographic-appearing fracture and reference flat traces using a thin lead pencil and straight edge such as drafting triangle. Measure the angle between each fracture trace and the reference flat trace for each wafer using a protractor.

Data Analysis

1. For the wafer fragments, label at least two (2) instances of non-crystallographic fracture if it occurred.

2. Impose each crystallographic fracture plane trace, correctly oriented, on a separate appropriate (Instructor Note 9) standard stereographic projection using the corresponding angular measurement relative to the reference flat trace.

3. Construct a diametral line that is orthogonal to each fracture plane trace. The constructed line should intersect or be in close proximity to a number of Miller indices that appear on the standard projection. Any of these indices then are for poles of planes (i.e., plane normals) that could explain the observed fracture. The field of candidates can be immediately reduced, roughly in half, by visually determining the inclination of the fracture plane and discarding those poles on the side of the projection that are not appropriate.

4. Refer to fracture plane observations reported in the literature [5,13-16] to determine whether any of the other remaining plane poles are consistent.

5. **Optional Activity:** Obtain a photomicrograph of the end view of the fracture plane and perform a second trace analysis to determine the exact identity of the plane in question, thus eliminating the other remaining candidates.

SAMPLE PLOTS AND DATA SHEETS: All of the experimental steps were performed twice to verify that the results were reproducible.

Laue Spot Appearance: All of the spots were circular and without any evidence of asterism.

Wafer Orientation: A schematic of the Laue photograph for Wafer #2 that was analyzed appears in Figure 1 imposed on a (111) stereographic projection in which the orthogonal $[\bar{1}2\bar{1}]$ and $[\bar{1}01]$ directions are shown. Six (6) prominent zones and corresponding axes are identified, including $[\bar{1}01]$ (Zone A). The measured ϕ, α angles allowing the zone axes to be plotted are given in Table I. The polished surface was identified to be approximately 4^{o} off (111) as directly viewed. Rotating the stereographic projection this angular amount about the $\pm[\bar{1}01]$ direction provided a nearly exact match with the standard cubic (111) stereographic projection. This result is in perfect agreement with the crystallographic information provided by the manufacturer.

Non-Crystallographic Fracture and Crystallographic Fracture Plane Traces: The outlines of the fragments of Wafers #1 and 2 are given in Figures 2 and 3, respectively. Although not present in Wafer #1, examples of non-crystallographic fracture did occur in Wafer #2. The traces of fracture planes that appeared to be crystallographic are denoted in both figures. Included are the measured angles between the fracture plane traces and the reference flat trace. The crystallographic fracture plane traces and their orthogonal constructions appear in a series of standard cubic (111) stereographic projections (Figures 4-6). Traces 2 and 5 were not analyzed since they are the same as Trace 1 (Figure 4).

Fracture Plane Candidates and Literature Comparison: Referring to Figures 4-6, those plane poles with low indices (h, k, and $l \leq |3|$) that are intersected (either exactly or nearly) by the orthogonal constructions are listed in Table II. The poles are divided based on the inclination of their planes to the polished wafer surface, and the impossible poles are noted and eliminated from consideration. The single-trace analysis results are consistent with the well-known observation (e.g., References [5] and [13]) of {111} as the primary fracture planes for silicon. However, the results are unable to distinguish between {110} fracture that has also been reported [13-16]. As such, a two-trace analysis is necessary to resolve which plane is involved in the wafers used in this study.

Second Trace Analysis: A photomicrograph showing an end view of the cleavage fracture plane in Wafer #1 appears in Figure 7. The angle between this plane and the polished near (111) plane was measured to be 73^{o}. Placing (Figure 8) this fracture plane

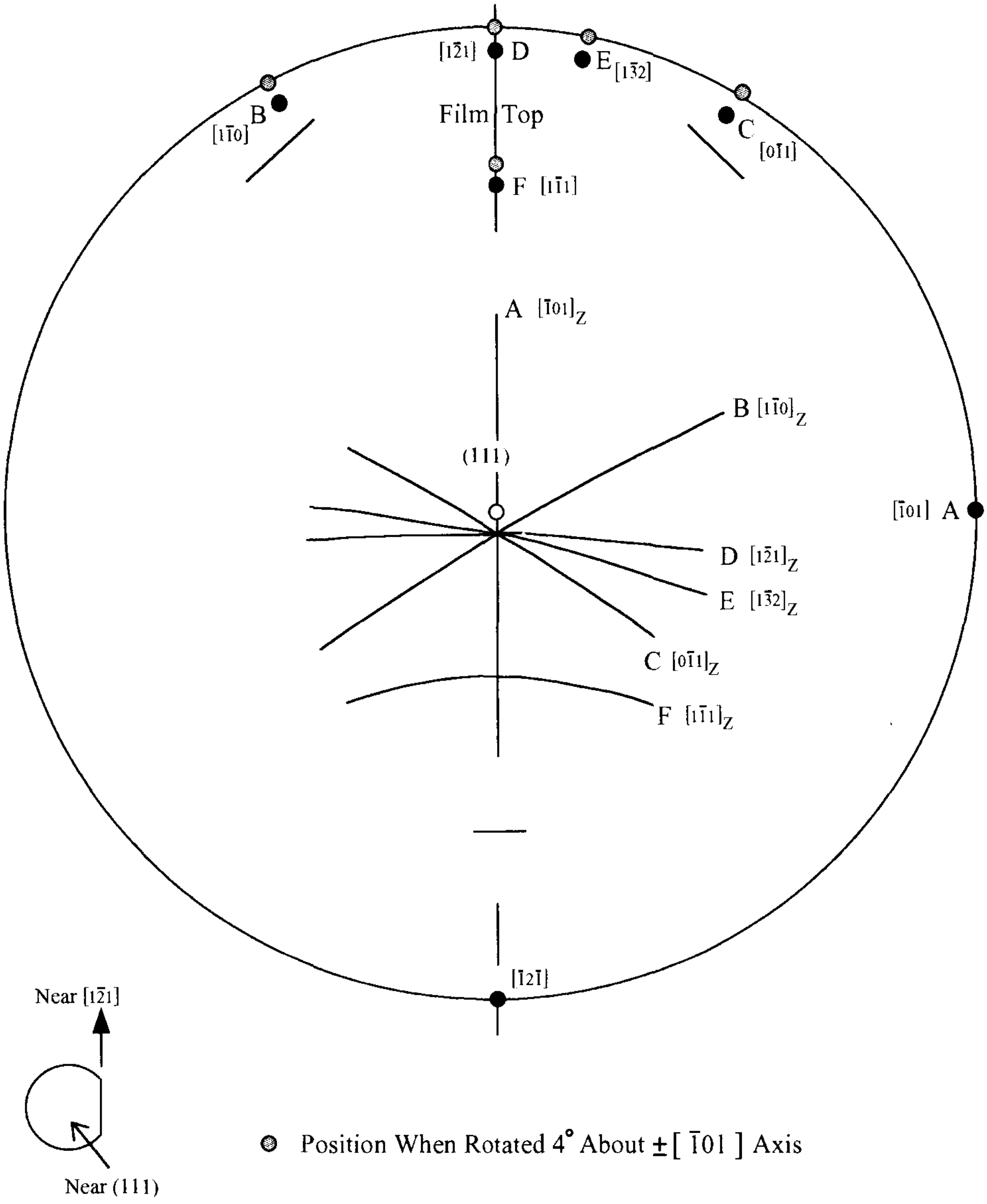

Figure 1. Back-reflection Laue photograph in combination with stereographic projection showing zones of diffraction spots and zone axes to identify the near (111) polished surface of Wafer #2 as directly viewed.

Table I. Measured Angular Information for Prominent Zones in Back-Reflection Laue Photograph of Wafer #2 (Figure 1)

Zone	Angle of Inclination (ϕ)	Azimuthal Angle (α)
A	0°	90° CCW*
B	4°	149° CCW*
C	4°	210° CCW*
D	4°	180° CCW*
E	$4^{+\circ}$	190° CCW*
F	$23^{+\circ}$	180° CCW*

---

*CCW = counterclockwise.

Table II. Candidate Fracture Planes Obtained from Single Trace Analyses (Figures 4-6)

Trace	Possible Planes	Eliminated Planes
1, 2, & 5	(233) (122) (133) (011) ($\bar{1}$33) ($\bar{1}$22) ($\bar{2}$33) ($\bar{1}$11) ($\bar{3}$22) ($\bar{2}$11)	(322) (211) (311) (100) (3$\bar{1}\bar{1}$) (2$\bar{1}\bar{1}$)
3 & 6	(323) (212) (313) (101) (3$\bar{1}$3) (2$\bar{1}$2) (3$\bar{2}$3) (1$\bar{1}$1) (2$\bar{3}$2) (1$\bar{2}$1)	(232) (121) (131) (010) ($\bar{1}$3$\bar{1}$) ($\bar{1}$2$\bar{1}$)
4	(332) (221) (331) (110) (33$\bar{1}$) (22$\bar{1}$) (33$\bar{2}$) (11$\bar{1}$) (22$\bar{3}$) (11$\bar{2}$)	(223) (112) (113) (001) ($\bar{1}\bar{1}$3) ($\bar{1}\bar{1}$2)

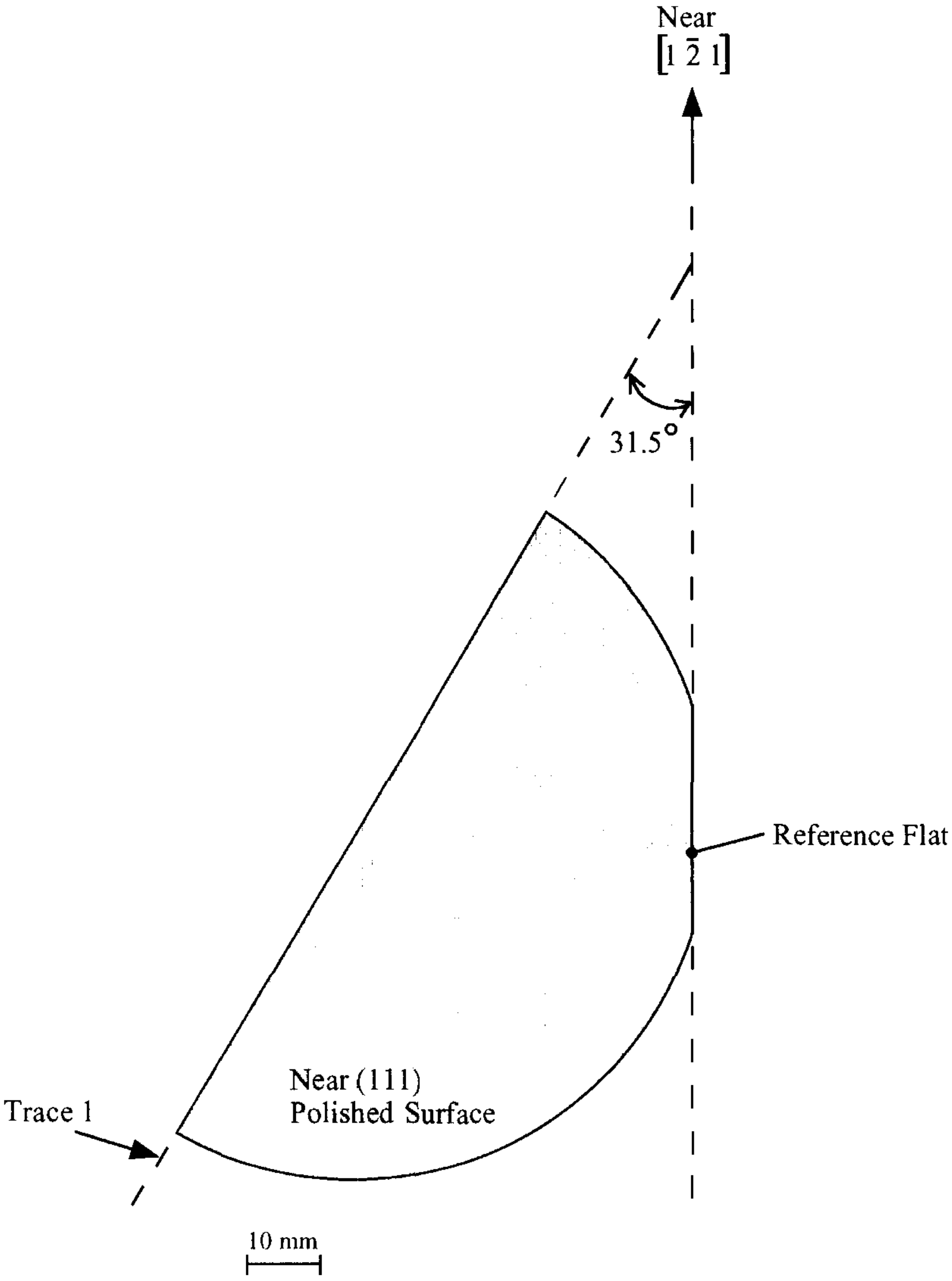

Figure 2. Wafer #1 fragment exhibiting cleavage fracture with associated Trace 1.

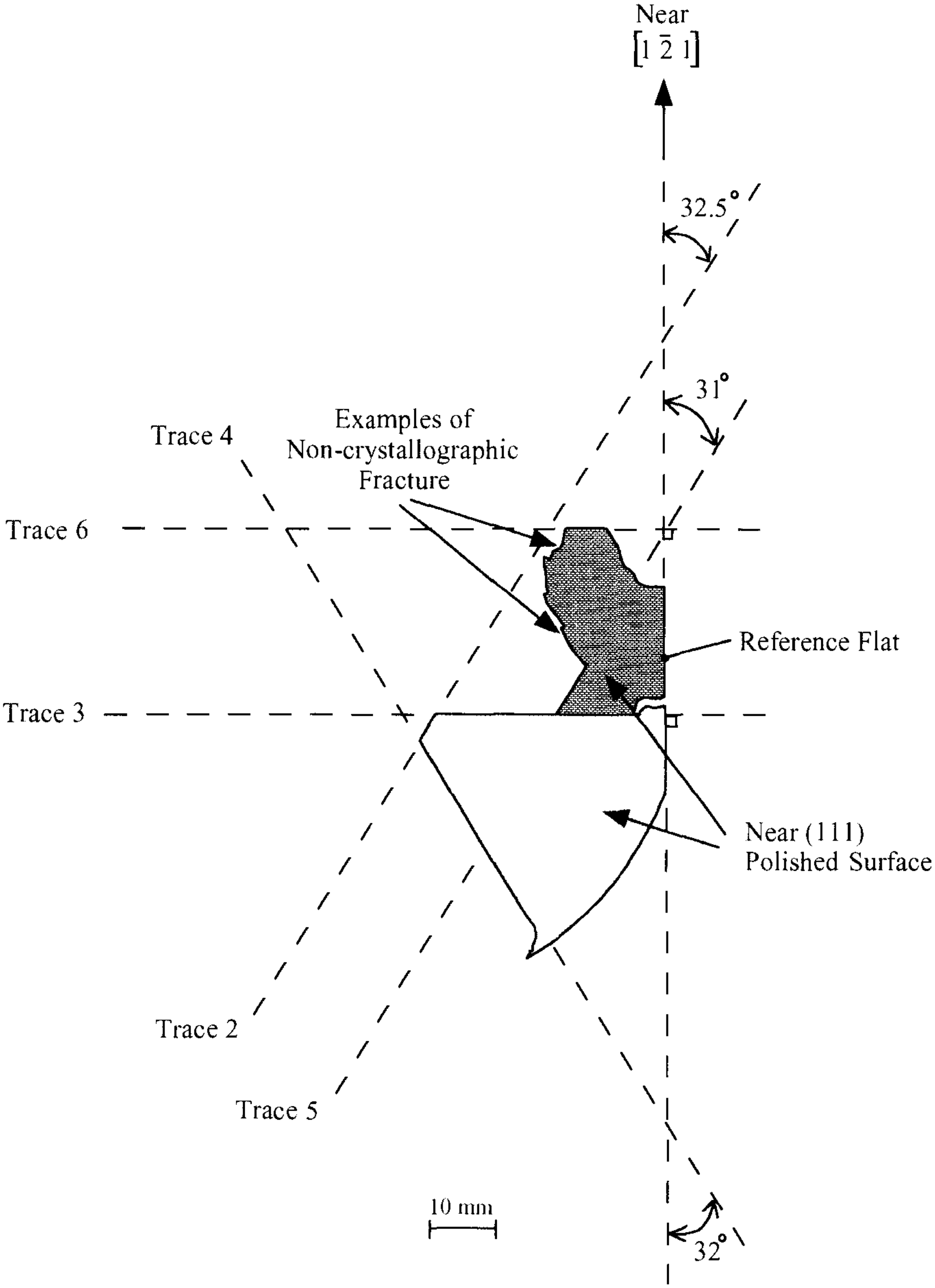

Figure 3. Wafer #2 fragments exhibiting both cleavage fracture with associated Traces 2-6 and non-crystallographic fracture.

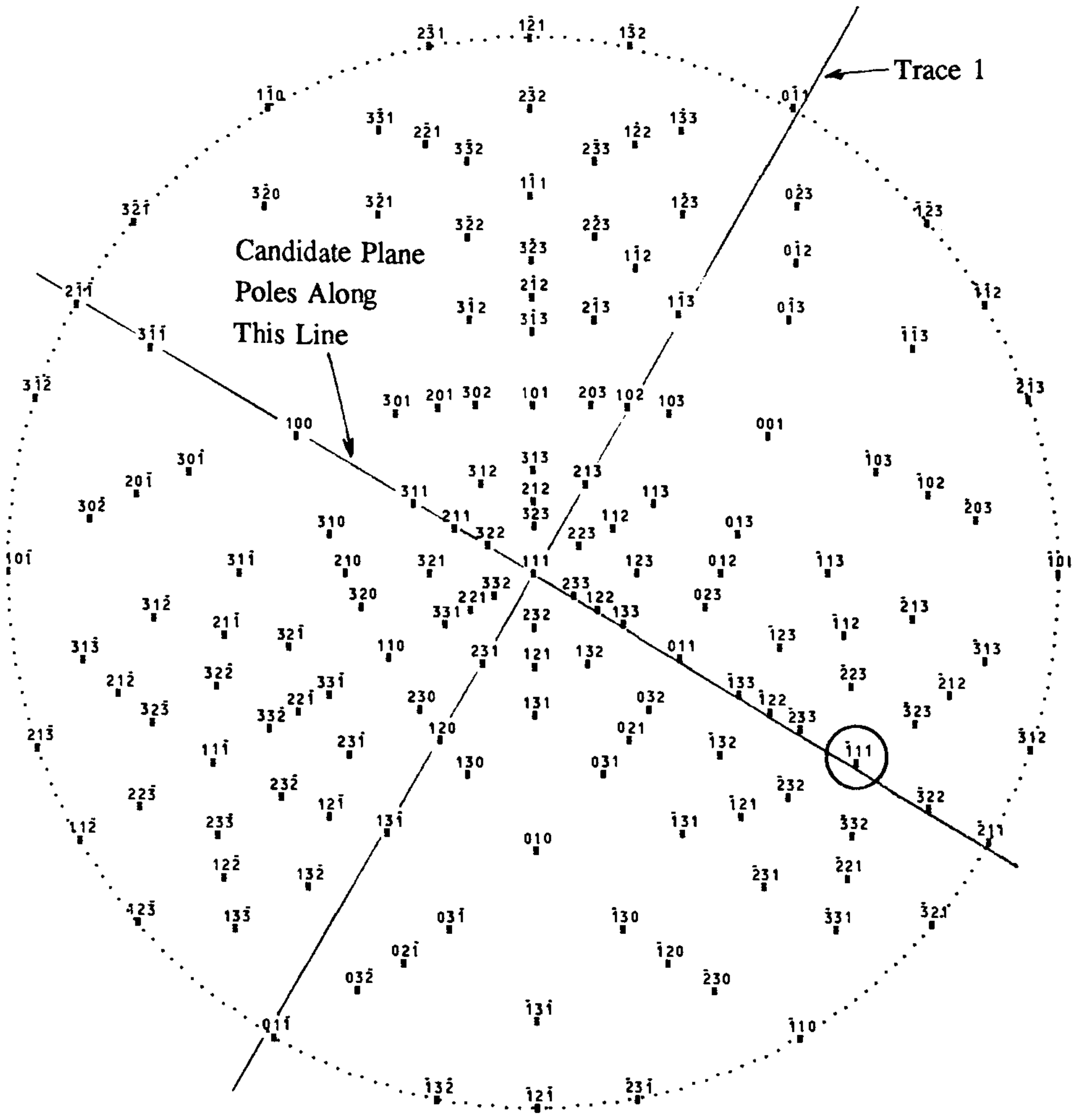

Figure 4. Standard cubic (111) stereographic projection showing Trace 1 and corresponding candidate fracture plane poles.

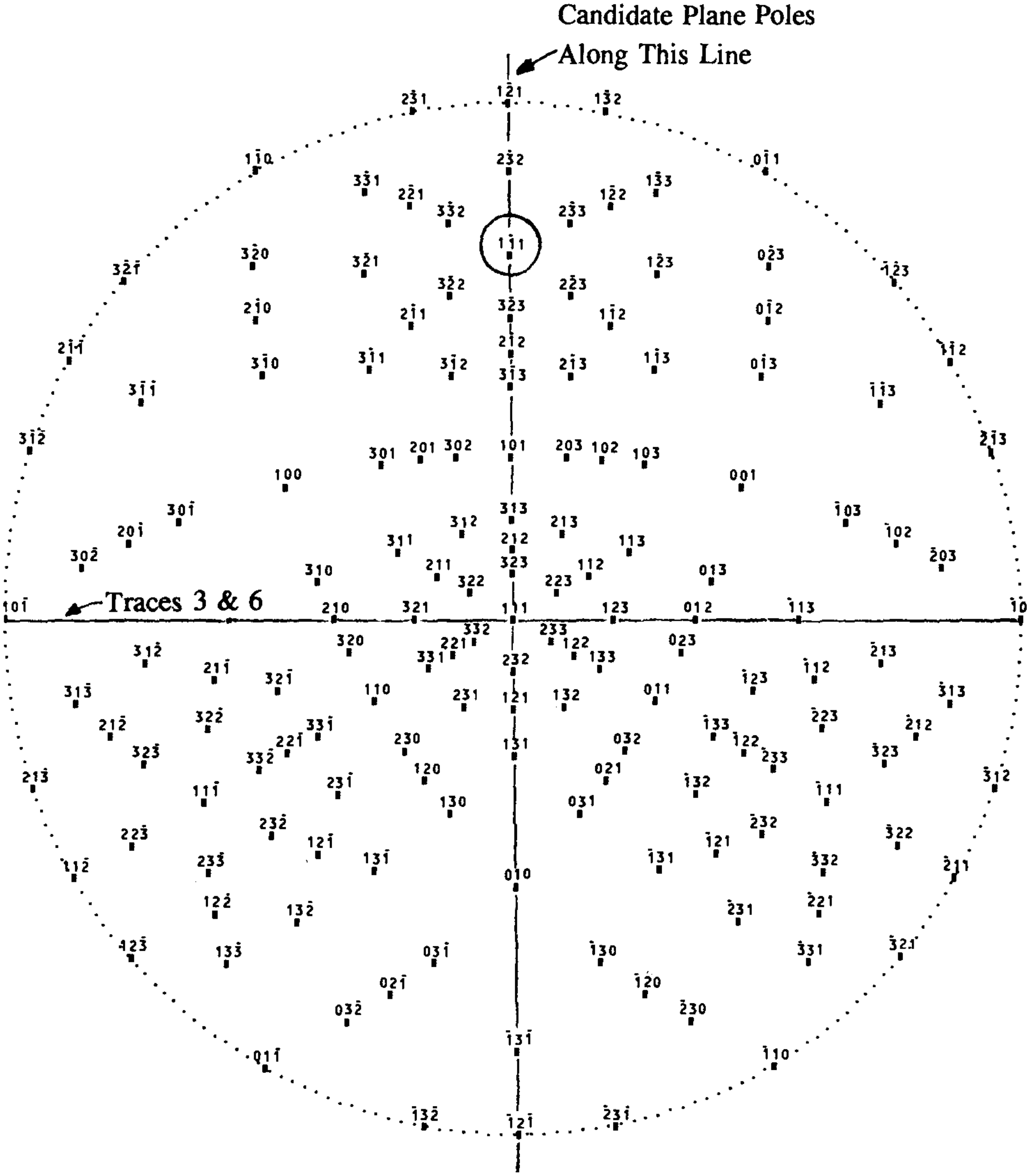

Figure 5. Standard cubic (111) stereographic projection showing Traces 3 and 6 and corresponding candidate fracture plane poles.

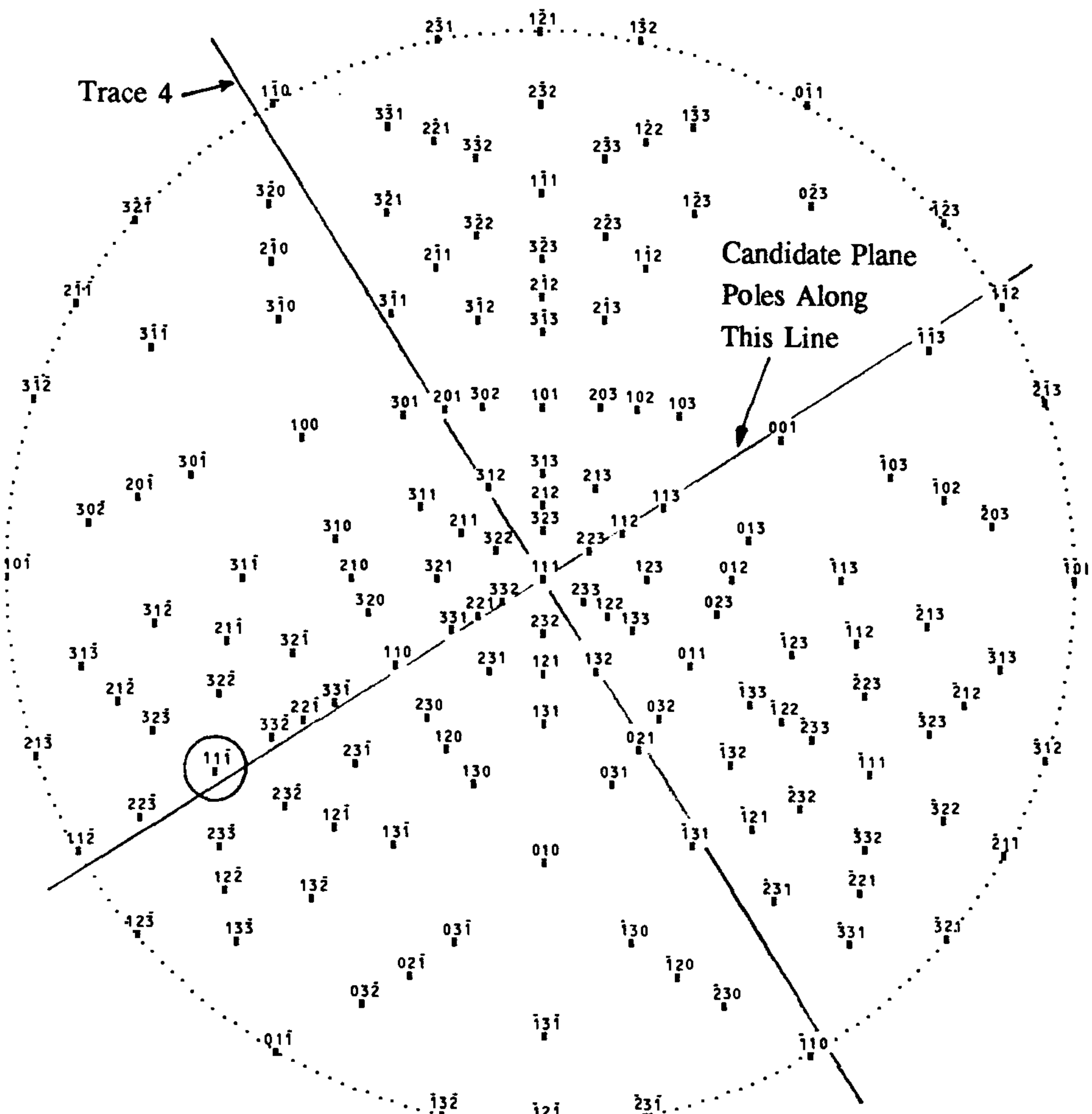

Figure 6. Standard cubic (111) stereographic projection showing Trace 4 and corresponding candidate fracture plane poles.

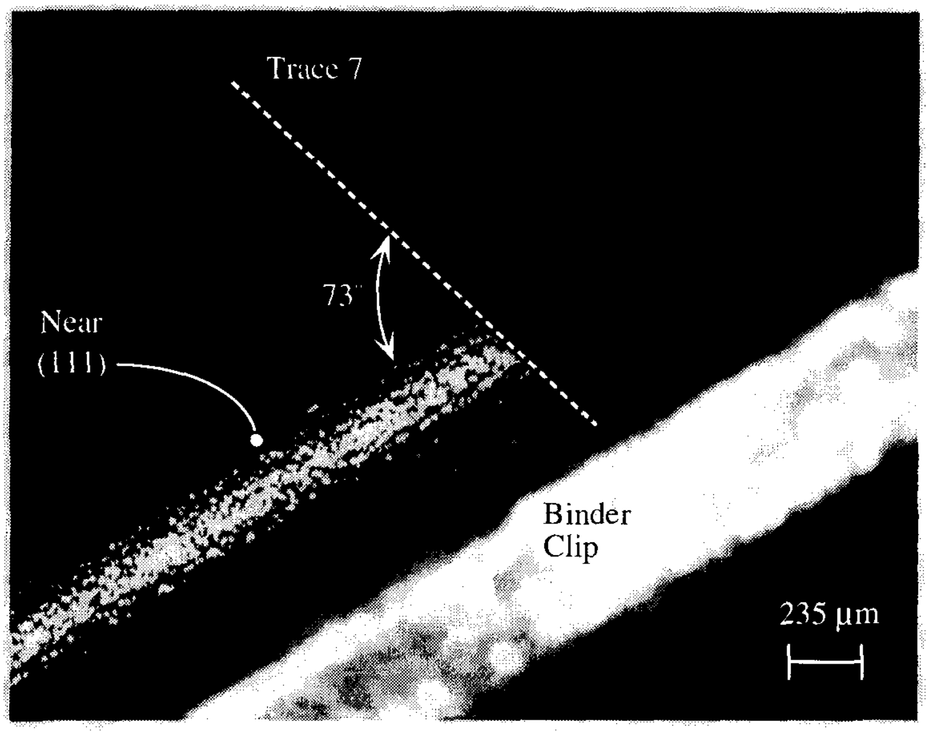

Figure 7. Photomicrograph of end view of Wafer #1 showing cleavage fracture with associated Trace 7.

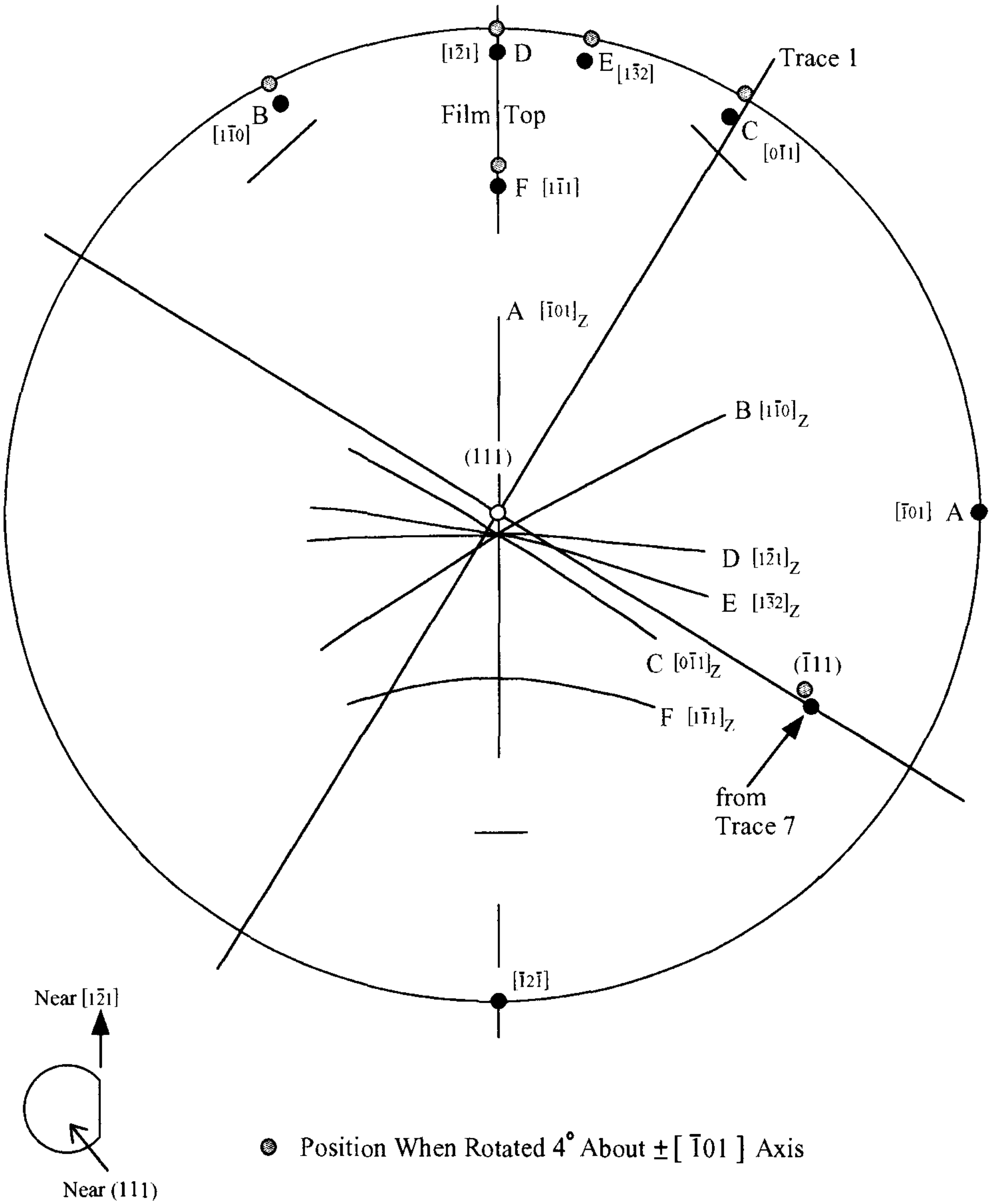

Figure 8. Trace 1 together with Trace 7 angle plotted on Figure 1 to identify $(\bar{1}11)$ cleavage fracture in Wafer #1.

trace on the stereographic projection appearing in Figure 1 and performing the necessary 4^{o} rotation about the $\pm[\bar{1}01]$ axis allowed the fracture plane to be identified as $(\bar{1}11)$.

INSTRUCTOR NOTES:

1. Stereographic projections and their applications are discussed in considerable detail by Johari and Thomas [17]. A number of very nice worked examples involving stereographic projections are presented by Moon [18].

2. If it is not possible to perform the Laue experiment, orientation information provided by the manufacturer can be relied upon, allowing the trace-analysis portion of the experiment to be performed still.

3. A small (1 cm x 1 cm) piece of double-sided tape (3M Scotch brand) was used to attach the wafer firmly to the goniometer and preserve the necessary orthogonality condition for the incident surface of the wafer and x-ray beam. The wafer should subsequently be removed as soon as is practical to lessen detachment difficulty that can lead to unwanted breakage.

4. In this work, the polished side of the wafer was as-viewed with the reference flat on the right, and care was taken to align the flat vertically.

5. **SAFETY PRECAUTION: Consideration should be given to obtaining the Laue photograph for the students ahead of time and simply showing the experimental set-up and explaining how the photograph was taken.** Using a copper x-ray tube, a high quality photograph resulted with Polaroid Type 57 film (positioned 3 cm from the wafer) when the generator was operated at 30 kV and 20 ma for an exposure time of 7.5 min. The time was sufficiently short that fluorescence was not a problem.

6. See companion paper [11] in this Workshop for availability of standard cubic stereographic projections.

7. The agreement between the plotted zone axes and the best candidate standard projection (i.e., (111) in this experiment) will be reasonably close but still only approximate since the wafers were intentionally sliced 3.5 to 4.5^{o} off of (111) by the manufacturer.

8. The fragments were obtained by fracturing as-received whole wafers in cantilever flexure by hand. Care was taken to align the wafer in the hand-held clamp so that the desired {111} fracture occurred preferentially. **SAFETY PRECAUTION: It is required that goggles be worn while wafers are being flexed to avoid the danger of flying fragments!** It is suggested that 75 mm diameter wafers be tried as they are much thinner and hence easier to fracture.

9. Since the surface incident to the x-ray beam was determined to be approximately 4^{o} from (111), the standard (111) projection is adequate.

REFERENCES:

[1] Barrett, C.; and Massalski, T.B.: Structure of Metals Crystallographic Methods, Principles and Data, Third Revised Edition. Pergamon Press, 1980, pp. 90-96.

[2] Cullity, B.D.: Elements of X-ray Diffraction, Second Edition. Addison-Wesley Publishing Company, Inc., 1987, pp. 149-160.

[3] Barrett, C.; and Massalski, T.B.: op. cit., pp. 30-50.

[4] Cullity, B.D.: op. cit., pp. 63-78.

[5] Lawn, B.R.; and Wilshaw T.R.: Fracture of Brittle Solids. Cambridge University Press, 1975, p. 17.

[6] "Silicon in Electronics," Excerpt from KEYNOTES '91-92. MEMC Electronic Materials, Inc., 501 Pearl Drive (City of O'Fallon), P.O. Box 8, St. Peters, Missouri 63376.

[7] Mayer, J.W.; and Lau, S.S.: Electronic Materials Science: For Integrated Circuits in Si and GaAs. Macmillan Publishing Company, 1990, p. 143.

[8] Barrett, C.; and Massalski, T.B.: op. cit., pp. 92-95.

[9] Cullity, B.D.: op. cit., pp. 158-160 and 260-265.

[10] Barrett, C.; and Massalski, T.B.: op. cit., pp. 211-222.

[11] Coyne, P.J., Jr.; Kohne, G.S.; and Elban, W.L.: PC Laser Printer-Generated Cubic Stereographic Projections with Accompanying Student Exercise, National Educators' Workshop: Update 94, NASA CP- , 1994.

[12] Barrett, C.; and Massalski, T.B.: op. cit., pp. 44-47.

[13] Misra, A.; and Finnie, I.: On the Scribing and Subsequent Fracturing of Silicon Semiconductor Wafers. J. Mater. Sci., vol. 14, no. 11, November 1979, pp. 2567-2574.

[14] Kaufman, M.J.; and Forty, A.J.: A Detailed Fractographic Analysis of Cleavage Steps in Silicon. J. Mater. Sci., vol. 21, no. 9, September 1986, pp. 3167-3172.

[15] Tsai, Y.L.; and Mecholsky, J.J., Jr.: Fractal Fracture of Single Crystal Silicon. J. Mater. Res., vol. 6, no. 6, June 1991, pp. 1248-1263.

[16] Tsai, Y.L.; and Mecholsky, J.J., Jr.: Fracture Mechanics Description of Fracture Mirror Formation in Single Crystals. Int. J. Fract., vol. 57, no. 2, September 15, 1992, pp. 167-182.

[17] Johari, O; and Thomas, G.: The Stereographic Projection and Its Applications. Techniques of Metals Research Volume IIA, Interscience Publishers, 1969.

[18] Moon, J.R.: Worked Examples in Stereographic Projection. Monograph No. 2, The Institution of Metallurgists, 1978.

SOURCES OF SUPPLIES (1994 Prices): Polished single crystal silicon wafers in several diameters are available from MEMC Electronic Materials, Inc., 128 Corporate Center, 70 Blanchard Road, Burlington MA 01803. The cost is nominally $12.00-15.00 each for 100 mm wafers. The Greninger chart and Wulff net, both plastic, are available from Polycrystal Book Service, P.O. Box 3439, Dayton, OH 45401-3439 at a cost of $20.00 each. Various standard cubic projections (18 cm diameter) can also be purchased from Polycrystal for $15.00 each.

ACKNOWLEDGEMENTS: The author is deeply indebted to D.W. Sloat, MEMC Electronic Materials, Inc., for providing the polished silicon wafers with crystallographic information that allowed the current work to be accomplished. The help of D.S. Richards, Dept. of Electrical Engineering and Engineering Science, Loyola College, in using computer graphics to generate Figures 1-3 and 8 is gratefully recognized. Special thanks to R.D. Eastman, Dept. of Computer Science, Loyola College, for using image processing software to obtain Figure 7 in its final form. It is highly unlikely that this work would have been performed had not R.W. Armstrong, Dept. of Mechanical Engineering, University of Maryland, College Park, masterfully demonstrated the use of stereographic projections to the author to solve a variety of applied crystallography problems.

PAPER CLIP FATIGUE BEND TEST

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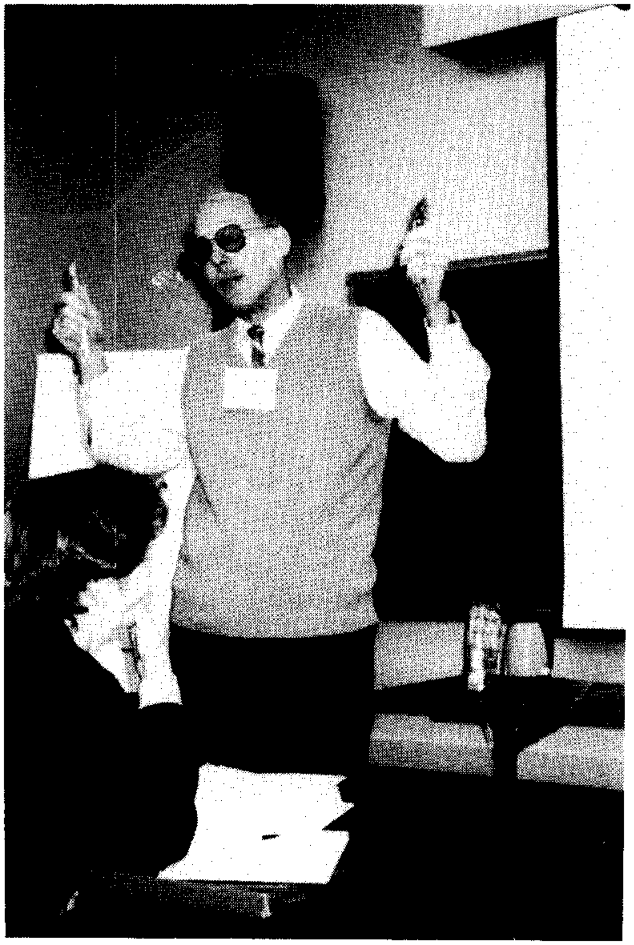

Paper Clip Fatigue Bend Test

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KEYWORDS: Fatigue Test, Wire, Paper Clips

PREREQUISITE KNOWLEDGE:

The student should be familiar with different types of paper clips and may be aware that paper clips can be made of materials other than steel wire. The student is most likely familiar with the fact that a paper clip can be broken or fatigued by bending it back and forth, too.

OBJECTIVES:

Fatigue tests are performed to determine the effect of different bend angles on the response of a material to cyclic loading. A thin wire rod, such as found on the straight leg of a regular size paper clip, is selected and cut to three-quarters of one inch long. Each rod is then held and cycled through a bend angle of a specified size until the rod fails. This process is repeated three times for a specified bend angle. The average number of cycles to failure for three samples is then calculated and plotted on a graph. To show how the number of cycles changes as the angle of bend changes four different bend angles are used. Next the sequence is repeated for a different size paper clip and the findings compared to illustrate the wire size effect and the impact of the fatigue attribute of paper clip wire.

EQUIPMENT AND SUPPLIES:

1. Paper Clips. Regular size or Associated Paper clips No.1 (H4-02001) and Jumbo, Associated Paper Clips, Jumbo (H4-2002)
2. Protractor made with an 8 1/2 in. by 11 in. piece of paper, and a plastic protector page
3. Diagonal Cutting Pliers
4. Two pairs of regular Pliers or Homemade wire sample holders
5. Safety Glasses
6. Gloves
7. Wire gage measure or Vernier Caliper
8. Data Sheet see below for the format

PROCEDURE:

The first step is the creation of hand made symmetric paper protractor with total angles of 45, 90, 135 and 180 degrees designated on the paper. This protractor is designed so that a total angle will be half of its degree displacement to the left and the other half to the right of the central position of the starting position for a test. A full cycle is a left off-set followed by a center pass through to a right off-set and return to the center.

The second step is to make four trials, each at a different bend test angle. A trial is the processing of three samples at a selected bend angle. This means that for each bend test angle, three samples are made, cycled to failure, the number of cycles to failure recorded and the average number of cycles tallied. After the first trial the three remaining test angles are to be processed, and then prepare a graph with labeled axes of the test angle on the vertical axis against the number of cycles to failure on the horizontal axis. The line on the plot should be labeled "Regular". The plot should be titled "Fatigue Bend Test for Paper Clips", and you should add where the work was done, your name and the date.

The third step is the selection of a second size of wire such as that found in a jumbo paper clip and making trials. Again, four trials, each with three samples and the determination of an average number of cycles to failure for each of the four test angles listed above, is required. The findings are plotted on the same graph as for the regular size paper clip and labeled "Jumbo". A comparison between the jumbo and regular paper clip test results can show the impact of size on the 'fatigue curve'.

PAPER PROTRACTOR:

To make a **Paper Protractor** secure an 8.5 by 11 inch sheet of paper and fold it in half parallel to the long length. Select one narrow end of the folded sheet and fold back the end until the short end coincides with the long fold line to obtain a 45 degree angle. Repeat this folding with the 45 degree folded portion of the sheet so a 22.5 degree angle is obtained. Repeat the folding sequence for the other half of the sheet. Unfold the sheet and draw a dark line to specify and dimension the 45, 90, 135, and 180 degree included angles that are symmetric to the long 11 inch fold line. Darken the longest fold line on the protractor as this line should be *in line* with the extended part of the fastener when a test is run.

Hint: Place a clear plastic protector page (an overhead transparency is suggested) over the Paper Protractor and use masking tape around the edge to secure the clear cover page to the protractor page.

SAMPLE PREPARATION:

Secure several regular sized paper clips and prepare a dozen rod pieces each three-quarters of an inch long by cutting the semi-circular loop ends from the paper clips. This process is to be repeated for the Jumbo paper clip, too.

TEST ACTIVITY, DATA AVERAGING AND GRAPHING:

Tests are to be performed to determine the effect of size of bend angle on the fatigue behavior of a material. This first set of trials is for the regular paper clips.

The initial step for trial one is to select the rod, grip each end of the rod in the jaws of a pair of pliers being sure the rod is fully inserted 1/4 inch into the jaws. Next place the nose of one pair of the pliers with the sample extending over the protractor with the plastic protection page at the vertex of the protractor and in line with the center line of the protractor. There should be a 3/8 inch long open length that covers the protractor, and in which the bending can occur. The second pair of pliers will be on top of the plastic protection page.

When the 45 degree test angle is chosen, the cycle of bending starts at the center line, moves to the left, moves to the right passing the center line, and returns to the centeral position. Count this as one complete cycle. This cycle is repeated until the saample fails at which time the number of full and partial cycles are counted and recorded in the data sheet show in Table 1. Several practice runs are recommended to develop skill in holding each end of the rod securely while bending the rod.

Please fasten the protractor onto a surface with masking tape and then hold one end of the rod at the origin on the protractor and move the pliers back and forth within the appropriate bend angle lines.

Remember when you start bending that the axis of the rod should be in line with the centerline of the protractor. For each bend angle three samples are used, loaded to failure, and the number of cycles for each sample to fail recorded, and the average number of cycles to failure tallied. This completes trial one.

For larger rods the use of a prefabricated holder instead of pliers is recommended. See the Appendix for specifications. THE EXPERIMENTER SHOULD TAKE CARE TO WEAR SAFETY GLASSES and to carefully watch the rod to discern the first site on the rod where failure starts. The region will discolor,change in geometry, and fracture. Sometimes the experimenter can feel a change in the resistance offered while bending, too.

Trials two through four follow the same sequence, except that the angle is changed to 90 degrees, to 135 and to 180 degrees. Prepare a graph of the bend angle on the vertical axis against the number of cycles to failure on the horizontal axis. Be sure to label the axes with a name and scale . Each point placed on the graph should be placed on the plot and circled by a symbol that surrounds the data point. Connect the symbols on the graph sequentially from high to low by using the data points to 'line-up' the data points, but drawing a line ONLY between the symbols. This process lets the data points be clearly seen. Be sure to label the line "Regular" for the regular paper clip. To complete the graph add on the title "Fatigue Bend Test for Paper Clips", the place where you did the testing, your name, and the date.

Table 1. Regular Paper Clip Data Table

Regular Paper Clip				
Bend Angle	45	90	135	180
Trials	Cycles	Cycles	Cycles	Cycles
Sample 1				
Sample 2				
Sample 3				
Total				
Average				

Run a second set of trials for the jumbo paper clip rods repeating the same four trials used for the regular paper clip wire rods. This includes making three sample runs at a specified bend angle, averaging the data to place in Table 2, below and adding the results to the graph. The label "Jumbo" should be attached to the curve drawn by connecting the 'Jumbo average points symbols" with a series of sequential lines. The symbol could be a triangle with the data point inside.

Table 2. Jumbo Paper Clip Data Table

Jumbo Paper Clip				
Bend Angle	45	90	135	180
Trials	Cycles	Cycles	Cycles	Cycles
Sample 1				
Sample 2				
Sample 3				
Total				
Average				

A comparison between the jumbo and regular paper clip can show the impact of size on the 'fatigue curve'. Features to notice include how rapidly the curve 'falls' as the size to the total angle changes. Remember that the size of the bend angle represents the amount of mechanical straining introduced by the experimenter. The larger paper clip might have a curve that is below the 'regular' paper clip because it is larger in diameter and less ductile or bendable.

COMMENTS:

There are many opportunities that can be developed from this basic exercise.

1. For example a paper fastener usually made of brass can be used instead of the paper clip. In this case two sizes of paper fastener can to be studied: Brass Round Head Fasteners No. 6 and No. 4. Two experiments are to be made. The first is for the No. 6 Paper Fastener. Three samples at each of four bend angles are to be evaluated for experiment one. Follow the same procedure outline for the paper clips with recommended angles of bend of: 45, 90, 135, and 180 degrees. This time align the centerline of the protractor with respect to an axis in which the paper fastener is mounted. It may be helpful to use just the paper protractor without the plastic cover page mounted with masking tape.

To mount the paper fastener in a parallel jaw clamp or a vise, separate the legs of the fastener and secure the free end of one leg in the clamp mid-way along its length. Take the extended portion of the paper fastener between your fingers and bend the fastener back and forth perpendicular to the width of the leg through either a 180, 135, 90, or 45 degree angle, counting the number of complete bend cycles to failure. Record this value in the data table. Complete the remaining trials. Each trial has three tests for a bend angle, an average value calculated for the number of cycles to failure as shown in the table, and a plot of the average values on a graph against the total bend angle. Be sure to label the curve, the axes, the graph with a title, where the work was done, your name and date.

The second experiment is for the No. 4 Paper Fastener. Be sure to place the findings from Paper Fastener No. 6 with a labeled curve on the same graph as for Paper Fastener No. 6. To help size the bend angle a **Paper Protractor** as presented above, can be made. Also, to save Paper Fasteners and use all available legs, the second leg of the fastener can be used for the next test to reduce the number of fasteners destroyed.

2. A comparison between the jumbo and regular paper clip can show the impact of size on the 'fatigue curve'. The same is true for the Paper Fastener. Additional experiments with different materials such as rod samples of aluminum, brass, welding rods, etc., and some plastics can add depth of understanding as well as provide interesting contrasts on a plot of results. In all cases the material and diameter of the rod should be noted on the graph. For the future different types of (heat) treating of the rods can be considered as well as changing the surface condition by abrading with emery clothes of various grits or even adding a notch. The latter case provides for the calculation of a notch sensitivity ratio when a base test is for an un-notched rod. Another variation is the cleaning of the surface of the rods with different mediums such as acetone, oil, blueing, and other operatives, and each time creating sensivitity ratios to a 'base' rod condition.

3. To assist the reader and give confidence in the performance of Fatigue Bend Tests the following data is supplied. First, an aluminum CX WELD NO. 23 filler rod (diameter 0.125 inches) was used, and second, a gas welding bronze colored steel filler rod (diameter 0.091 inches) was tested. Figure 1 show the graphical comparison.

Table 3. Aluminum Filler Rod Data

Aluminum Filler Rod: CX WELD NO. 23				
Bend Angle	45	90	135	180
Trials	**Cycles**	**Cycles**	**Cycles**	**Cycles**
Sample 1	8	3.5	2	.75
Sample 2	8	3.5	1	.50
Sample 3	7.5	2.5	1.5	.50
Total	23.5	9.5	4.5	1.75
Average	7.8	3.2	1.5	.6

Table 4. Steel Filler Rod Data

Bronze Colored Steel: Welding Filler Rod				
Bend Angle	45	90	135	180
Trials	Cycles	Cycles	Cycles	Cycles
Sample 1	38	7.5	4.5	2.75
Sample 2	37	8	4.75	3.25
Sample 3	43	8	4.75	2.75
Total	118	21.5	14.0	8.75
Average	39.3	7.2	4.7	2.9

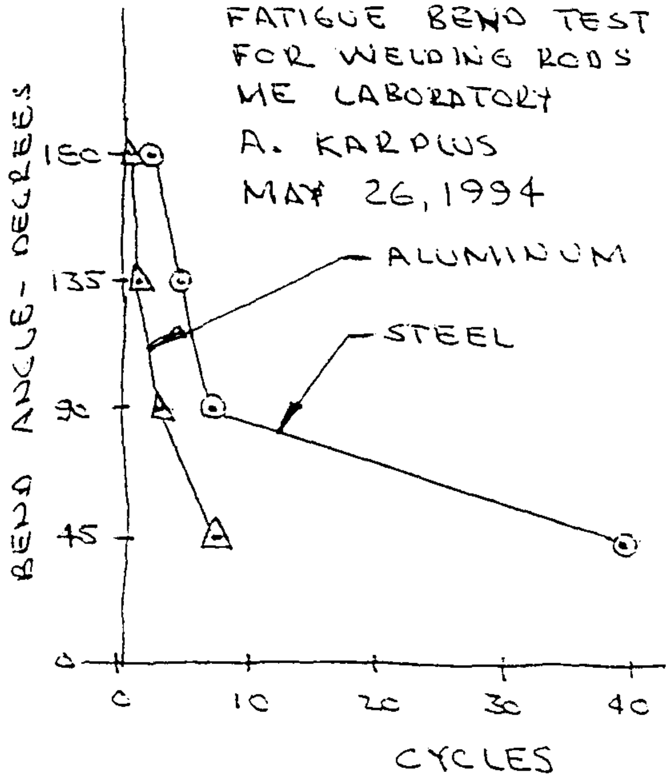

Figure 1. Sample Fatigue Curve (Data from Tables 3 and 4)

4. The use of holders is best for wire rods that have diameters for which tooling to make the holes in the ends of the holders, exists. Diameters smaller than 0.040 inches is often a machine shop limit. If the hole prepared is larger than appropriate, the wire can 'flip around', easily come out of the holder(s), and not bend. This offers the opportunity to experiment with longer and shorter than 3/4 of an inch rods to learn the best ways to hold a rod to be bent.

APPENDIX : Holders

A rod sample holding tool set can be prepared by seccuring a pair of rods such as 3/8 inch diameter round, square, or hexagonal cross-section stock each five inches long. Next machine a hole of the proper diameter (check the rod size to be tested) with a 0.001 to 0.002 inch clearance one quarter to one- half inch deep in one end of each of the holders. Consider facing the end first to have a clean surface on the holders. Remember the wires under test must be long enough to clear the holders in the 180 degree bends (two half inch deep holes plus 3/4 inch bend region needs a one and three-quarters inch long bend sample). Also recall that diameters larger than 0.040 inches are available as drill sizes so that another approach may be best for smaller wire rods. Regular pliers and even vise grips can be considered.

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Alan K. Karplus is Professor of Mechanical Engineering at Western New England College, Springfield, Massachusetts. He has a Bachelor's degree from Tufts College, a Master's degree from Iowa State University and a Ph.D. from Colorado State University. He has been involved with the freshman engineering program, coordinates the junior and senior mechanical engineering laboratory program, teaches Materials Science and supervises M.E. Senior Projects. He is a member of ASME, ASEE and ASM International.

MATHEMATICS FOR ENGINEERING MATERIALS TECHNOLOGY EXPERIMENTS AND PROBLEM SOLVING

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Mathematics for Engineering Materials Technology Experiments and Problem Solving

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KEY WORDS: Analysis, unit conversions, SI Units and U.S. Customary Units

PREREQUISITE KNOWLEDGE: Often students enter the first course in engineering materials technology with only basic algebra.

OBJECTIVES: To develop skill and confidence in applying mathematics to solving problems in engineering materials technology.

EQUIPMENT AND SUPPLIES:

Overhead projector calculator
Microcomputer

RATIONALE: Mathematics, when used wisely, can be a great asset in the study of engineering materials. This sentence defines the objective of this presentation to remind instructors to encourage their students to exercise this skill at every opportunity. All of us recognize that one cardinal principle of good teaching is to allow students to engage in hands-on activities as part of their learning. Not too long ago books dealing with engineering materials technology would include a statement such as: 'It is assumed that students have an adequate background in mathematics, physics, and chemistry for the level of treatment presented.' Is this statement an adequate description of the students coming into your classrooms today?

In spite of the general poor mathematical preparation of many of our students, it is doubly important to give them the opportunity, wherever possible, to use their limited knowledge of mathematics in their study of materials. In so doing they may experience, for the first time, how mathematics can be an excellent tool to use outside the math classroom in other studies. Second, if properly used, it can assist them in assimilating and mastering the principles of materials education.

Numerous examples throughout the study of materials can be found to provide students the experience, through mathematical calculation, to 1) determine, 2) predict, 3) estimate, and 4) analyze physical quantities. In this regard we must be sensitive to the requirement that students develop the habit of producing neat, accurate, and clearly written solutions to assigned problems. In that regard a statement of what the problem is and what is known about the problem, as well as a proper identification of the final solution WITH THE CORRECT UNITS INDICATED is part of the documentation.

ANALYSIS

Analysis, being a mental process, requires a database of knowledge along with the ability to apply that knowledge. Just as one cannot solve a cross word puzzle without a knowledge of words, so one cannot analyze a materials problem without a knowledge of materials and mathematics. This knowledge must be augmented by experience with the tools associated with these disciplines. The language of both engineering science and technology is mathematics. To express in words how, for example, forces acting on a material are related to each other, is difficult to describe with words alone but in mathematical terms this relationship can be expressed simply and easily.

In our just revised book, Engineering Materials Technology, Jim Jacobs and I have **not** assumed students bring to their class a strong mathematical background. We have tried to provide numerous aids in the book and instructor's manual that offer one stop shopping for problem solving, thereby enhancing the ease of understanding engineering materials with its associated mathematics. In the design of the book, we have made great use of work generated by you and our colleagues form previous NEW:Updates. The many experiments from NEW:Updates provide excellent opportunities for hands-on activates that help to make concrete demonstrations of important analysis techniques.

CONVERSION OF UNITS

In spite of repeated efforts to establish the world-wide system of units of measure known as SI (Internationale Systeme d'Unites) in the U.S. as the one and only legal system, the dual system of units that includes the U.S. Customary system still is practiced in many areas. Consequently, the conversion of units is still a necessary technique that must be practiced to perfection. In addition it is imperative that students of materials must realize the importance of substituting units as well as numbers into their formulas and other calculations in order to communicate effectively the results of their analyses in both systems of measure. Note this type of substitution in the illustrative problem below dealing with thermal conductivity.

We've devoted a segment of our Appendix to discussion of SI/US Customary Units and Conversions and included tables such as Names and Symbols of SI Units which lists most of the common derived units, a table with special names for some of these derived units.

Illustrative Problem

An insulating material placed inside the walls of a home is 6 in. thick. The heat flow is calculated to be 4.2 Btu/hr when the inside temperature is 70°F and the outside temperature is 0°F. Determine the thermal conductivity of the insulating material as the heat flow passes through 1 ft² of the wall. In addition to expressing k in units of Btu/(hr · ft · °F), convert your units to SI units using watts, meters, and Kelvin temperature.

Solution

$$k = \frac{q}{A} \cdot \frac{\Delta d}{\Delta t}$$

$$= \frac{4.2\ \text{Btu}}{1\ \cancel{\text{ft}^2} \cdot \text{hr}} \cdot \frac{6\ \cancel{\text{in.}}}{70°\text{F}} \left(\frac{1\ \cancel{\text{ft}}}{12\ \cancel{\text{in.}}}\right)$$

$$= 0.03\ \text{Btu}/(\text{hr} \cdot \text{ft} \cdot °\text{F})$$

Using conversion (Table A-7), we have

$$1\ \text{Btu}/\text{ft} \cdot \text{hr} \cdot °\text{F} = 1.729\ \text{W}/\text{m} \cdot \text{K}$$

$$k = \frac{0.03\ \text{Btu}}{\text{hr} \cdot \text{ft} \cdot °\text{F}} \left(\frac{\text{ft} \cdot \text{hr} \cdot °\text{F}}{1\ \text{Btu}} \cdot \frac{1.729\ \text{W}}{\text{m} \cdot \text{K}}\right)$$

$$= 0.052\ \text{W}/\text{m} \cdot \text{K}$$

Included in this special appendix on the SI system are some thirteen Rules for Usage of these units as seen on the next page. One example of an illustrative problem dealing with a computation of a tensile load and using the SI system of units is shown below. Again, it is stressed that this would be an unnecessary exercise if the US followed the rest of the world, with possibly one exception, and converted to the SI system of units in toto. Remember those wonderful fraction problems such as 3 5/8 + 2/3 = ? Wouldn't it be nice to avoid them?

Problem 1

Given: A metal rod under tensile load of 356 kN is allowed to withstand a unit stress of 110 NM/m².

Required: Find the diameter of the rod in millimeters.

Solution

1. Convert SI prefixes to powers of 10:

$$356\ \text{kN} = 356 \times 10^3\ \text{N}$$

$$110\ \text{MN}/\text{m}^2 = 110 \times 10^6\ \text{N}/\text{m}^2$$

2. Use the direct stress formula ($s = P/A$) and solve for the area (A):

$$A = \frac{P}{s} = \frac{356 \times 10^3\ \text{N}}{110 \times 10^6\ \text{N}/\text{m}^2} = 3.24 \times 10^{-3}\ \text{m}^2$$

3. Use the formula for circular area $\left(A = \frac{\pi}{4}D^2\right)$ and solve for diameter (D):

$$D = \sqrt{\frac{4}{\pi}(3.24 \times 10^{-3}\ \text{m}^2)}$$

or

$$D = \left[\frac{4}{\pi}(3.24 \times 10^{-3}\ \text{m}^2)\right]^{1/2}$$

$$\left[\frac{4}{\pi}(32.4 \times 10^{-4}\ \text{m}^2)\right]^{1/2}$$

$$= 6.429 \times 10^{-2}\ \text{m}$$

$$= 64.29 \times 10^{-3}\ \text{m} = 64.3\ \text{mm}$$ (rounding up and using three significant digits)

Rules for usage. For standardized usage, the following rules should be observed:

1. Uppercase (capitals) and lowercase letters are never interchanged: kg, not KG.
2. The same symbol is used for plurals: N, not Ns; 14 meters or 14 m.
3. No space is left between the prefix and its unit symbol: GHz, not G Hz.
4. To form products, a raised dot is preferred (or a dot on a line): kN · m, or kN.m. The dot may be dispensed with if confusion is not created by its absence.
5. To form quotients, one solidus (an oblique line), a fraction line (horizontal), or a negative power is used to express derived units: m/s, $\frac{m}{s}$, or $m \cdot s^{-1}$.

   Note: The solidus must not be repeated on the same line: m/s^2, not m/s/s. Also, $kg/(m \cdot s)$, $\frac{kg}{m \cdot s}$, or $kg \cdot m^{-1} \cdot s^{-1}$, but not kg/m/s. Note also the use of the parentheses to avoid ambiguity.
6. An exponent affixed to a symbol containing a prefix indicates that the multiple or submultiple of the unit is raised to the power expressed by the exponent.

   $$1\ mm^3 = 10^{-9}\ m^3, \text{ not } 10^{-3}\ m^3$$
   $$1\ cm^3 = 10^{-6}\ m^3, \text{ not } 10^{-2}\ m^3$$
   $$1\ cm^{-1} = 10^{2}\ m^{-1}, \text{ not } 10^{-2}\ m^{-1}$$
7. A period is used as a decimal marker. It is not used to separate groups of digits. A space is left for this purpose: 5 279 585 J, and 0.000 34 s.
8. Numbers are preferably expressed between the limits 0.1 and 1000, using the appropriate prefix to change the size of the unit: 5.23 GN.
9. For decimal numbers less than 1, the leading zero is never omitted: 0.625, not .625.
10. When units are written in words, they always start with lowercase letters except at the beginning of a sentence. If the unit is derived from the name of a person, the symbol is capitalized. Plurals of special names are written in the usual manner.

    125 watts or 125 W
    0.25 newton or 0.25 N
    58.6 hertz or 58.6 Hz
11. A space or hyphen may be used to form the product expressed in words: newton - meters or newton meters.
12. For quotients, the word *per* may be used: newton per meter squared, kilogram per cubic meter.
13. The kelvin (symbol K) is the standard unit of temperature. In writing this absolute temperature, the word *degree* or its symbol (°) is not used: 472 K. In addition, K may be used to express an interval or a difference in temperature. Celsius temperature is expressed in degrees Celsius with symbol °C. The unit degrees Celsius is equal to the unit kelvin and may also be used to represent an interval or a difference of Celsius temperature: 25°C. Temperature in K = temperature in °C + 273.15.

DIMENSIONAL HOMOGENEITY

Balancing of units in an equation is known as dimensional homogeneity. A simple example is the formula (a mathematical rule that can be expressed by the proper signs and symbols) for the area(A) of a rectangle, i.e. A = b×h where b represents the base and h the height. The units for A must balance or be the same as the product of b × h. Failure to evaluate an equation for substitution of the correct units that accompany the numbers assigned to each variable in the equation is the downfall of many neophyte analysts. The Illustrative Problem on torque, printed below, shows an example of this substitution.

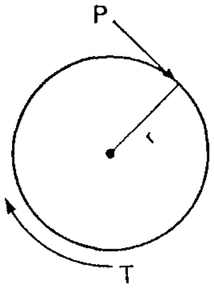
Illustrative Problem

How much torque *(T)* is needed to produce a shear stress of 79.6 MPa on the surface of a 40-mm-diameter shaft with a polar moment of inertia *(J)* equal to 25.12×10^{-8} m^4?

Solution

$$T = Pr \qquad \tau = \frac{Tr}{J}$$

$$r = \frac{D}{2} = 0.02 \text{ m} \qquad T = \frac{\tau J}{r}$$



$$T = P \cdot r$$

$$r = \frac{D}{2} = .02 \text{m}$$

$$= \frac{79.6 \text{ MPa} \times 25.12 \times 10^{-8} \text{ m}^4}{0.02 \text{ m}}$$

$$= \frac{79.6 \times 10^6 \text{N}/\cancel{\text{m}}^2 \times 25.12 \times 10^{-8} \text{m}^{\cancel{4}2}}{0.02 \cancel{\text{m}}}$$

$$= 999.8 \text{ N} \cdot \text{m} \cong 1 \text{ kN} \cdot \text{m}$$

This is a common practice when using the many formulas found in textbooks and other reference works in which constants are expressed in terms of a number.

A classic example taken from a reference on machine design using U.S. Customary units is the formula for calculating the torque on a round circular shaft transmitting a certain amount of power at a particular speed.

$$T = 63\,000 \text{ P/n}$$

The constant 63 000, is the key that should remind students that this formula requires that the correct units for both power (P) and speed (n) be used and no other. In this case the correct units are horsepower and revolutions per minute with the result expressed in pound inches. Incidently the student should be required to verify the value of the constant - 63 000. Further emphasis should be placed on insisting that students substitute the units as well as the numbers for every variable/term in an equation to ensure the proper units are arrived at in expressing the answer to their computations. An example taken from the materials literature is the expression for thermal shock resistance.

Thermal Shock Index(TSI) is a ratio of four variables. A complete discussion of TSI was provided by L.Roy Bunnell of Pacific Northwest Laboratory and can be found in NEW: UPDATE 1991. This figure of merit is an excellent tool for selecting ceramics with good thermal shock characteristics. Regardless of what system of units are employed the TSI is expressed using a standard set of units. If the units are mixed between the two systems of units the results are useless. This ratio is an excellent opportunity to exercise the reader's knowledge of how each of the four variables affect the resultant index.

$$\mathrm{TSI} = \frac{\sigma \times k}{\alpha \times E}$$

where

σ = tensile strength
k = thermal conductivity
α = linear coefficient of thermal expansion
E = modulus of elasticity (Young's modulus)

THERMAL SHOCK INDEX (TSI) FOR SELECTED MATERIALS

Material	σ (MPa)	k(W/cm · °C)	α(°C^{-1} × 10^{-6})	E (GPa)	TSI[a](W/cm)
Fused S_iO_2	68	6×10^{-2}	0.6	72	94
Al_2O_3	204	3×10^{-1}	5.4	344	33
Graphite	8.7	1.4	3.8	7.7	416
Soda-lime silica glass	69	2×10^{-2}	9.2	68	2.1

[a] TSI units in the U.S. Customary system are Btu · in./hr/ft^2; in the SI system they are W/cm.

Illustrative Problem

Determine the TSI for aramid fiber and E-glass fiber and indicate which has the best thermal shock resistance based on these calculations. Selected properties for each of these materials are provided in the table that follows:

Aramid fiber	E-Glass fiber
$E = 18 \times 10^6$ psi	$E = 10.5 \times 10^6$ psi
$\sigma = 400 \times 10^3$ psi	$\sigma = 500 \times 10^3$ psi
$k = 3.5$ Btu · in./hr · ft^2 · °F	$k = 7.0$ Btu · in./hr · ft^2 · °F
$\alpha = 0.8 \times 10^{-6}$ °F^{-1} (long.)	$\alpha = 1.6 \times 10^{-6}$ °F^{-1}

Solution

$$\mathrm{TSI}_{\text{aramid}} = \frac{4 \times 10^5 \times 3.5}{0.8 \times 10^{-6} \times 18 \times 10^6} = 9.7 \times 10^4 \text{ Btu} \cdot \text{in./hr} \cdot \text{ft}^2$$

$$\mathrm{TSI}_{E\text{-glass}} = \frac{5 \times 10^5 \times 7}{1.6 \times 10^{-6} \times 10.5 \times 10^6} = 2.1 \times 10^5 \text{ Btu} \cdot \text{in./hr} \cdot \text{ft}^2$$

Aramid fiber was the greater and therefore the best shock resistance.

ASSUMPTIONS

One of the first steps in the analysis of a problem is that of making simplified assumptions. If this step is done successfully what remains is what one believes are the determining factors. In essence these assumptions not only simplify the problem but in many cases make a tentative solution possible. The assumptions made are not forgotten but must be addressed in later considerations to evaluate their affect, if any, on the solution. As one of the final steps the assumptions set forth in the problem must be verified usually by conducting experiments to test their validity. Examples of simplified assumptions are: the internal structure of a 20 ft steel I-beam is homogeneous; the density of a solid ceramic body is uniform throughout; a surface is flat; a solid material under load is rigid; or the shear stress increases in a linear fashion outward from the neutral axis to the external surface of a shaft under torque (as seen in figure 4-28 below).

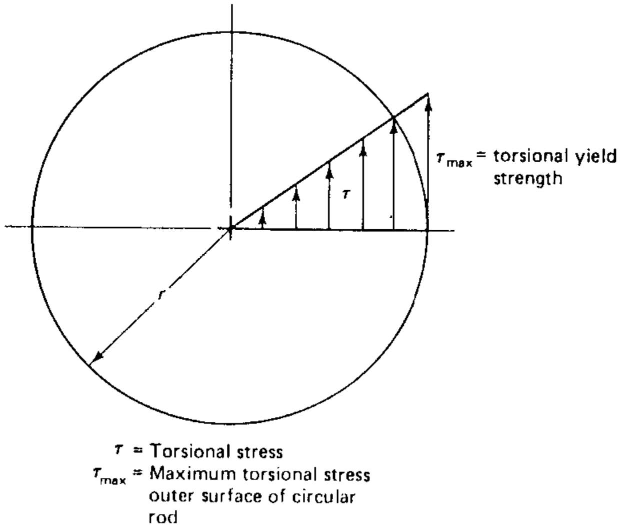


Figure 4-28 Distribution of torsional stress in a circular rod

A model is a representation of a mental picture of a problem just as a diagram represents a play in a football game. A simple sketch of an atom is a model that helps one understand the inner workings of atoms. An equation or formula is another example of a model which visually represents a problem. Other examples are charts, functional plots, and diagrams (free body diagrams, block diagrams, etc.). Functional charts or plots show relationships between variables. All of these are used throughout the study of materials as evidenced by the various figures used in this presentation. The use of a model and the mathematical equations that relate to it describe simply and easily the precise 'physical theory' of the situation. Using Figure 4-3, note the assumptions made but not specified in the construction of the model. Using a model and its accompanying mathematical relationships, the mental process of analysis is used to calculate the behavior of materials under various conditions of stress. This step in the process of engineering design illustrates the need for all involved in the world of materials to gain expertise in applying he tools of mathematics to every day problems.

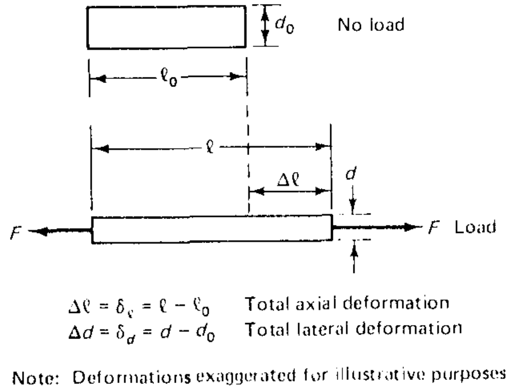


Figure 4-3 Rod under a tensile stress

STATISTICS

The result of analysis of materials using the tools of mathematics rarely represents exactly the physical behavior of 'real materials and devices'. Recall our discussion of assumptions. Further, the introduction of errors plus the fact that all measurements rarely yield the same value add to the fact that answers arrived at by whatever means are not true or exact. Consequently, the result is, at best, a statistical average. The results of the analysis of most problems take the form of a set of data. In other words the true magnitude of a property or quality of a material is a distribution function that requires statistical measures for its solution. The task now is to obtain the 'best value' of all the data or to extract the most useful information. To do this statistical data reduction procedures are used. Such procedures as determining significant figures (fictitious precision), or the accuracy of measuring instruments employ to a degree the tools of statistics. Production inspection of manufactured parts, quality control, and sampling are other areas that are based upon statistical measures. In the field of failure analysis, the concept of probability must be further addressed to properly document the results of such analysis. Because of the usual large amount of data involved, the computer is the tool to use to analyze it. H.T.McClelland, University of Wisconsin addresses this subject in his paper "Introduction to Usable Statistical Methods ", found in NEW:Upate 93.

COMPUTERS

An essential requirement in today's technological world is to be computer literate. Without an understanding of the digital computer, its role, and its use, it is most difficult if not impossible for a person to function in today's age. In addition to the computing power of a computer, the use of computer spread sheets makes available most mathematical functions and statistical quantities needed for calculation, searching tabular data, iterative problem solving, and the display of numbers graphically for better visualization of the data. A review of NEW:Update 93 discloses that of 39 experiments described 9 (or about 25%) specifically mentioned the use of a computer in conducting the experiment. One contributor mentioned prerequisite knowledge of the basic application of the microcomputer and DOS commands as being essential ("Graphing Techniques for Materials Laboratory Using Excel" by Nikhil K. Kundu, Purdue University). The addition of automated data collection system to the Charpy Impact Tester allowed a single student to conduct the experiment in half of the time previously required with a ten-fold increase in the accuracy of the measurements. ("An Automated Digital Data Collection and Analysis System for the Charpy Impact Tester" by Glenn S. Kohne and F. Xavier Spiegel, Loyola College).

SUMMATION

A working knowledge of mathematics and microcomputers, plus the ability to apply it to engineering materials technology experiments and other problem solving, permits one to more efficiently learn new knowledge of materials and to apply that knowledge for ones own benefit as well as for other members of our advanced technological society. Recognizing that students will need assistance in improving their competencies in mathematics will enable instructors to improve engineering materials technology teaching.

REFERENCES

1. Jacobs, James A. and Thomas F. Kilduff. ***Engineering Materials Technology,*** 2nd Edition, Prentice-Hall, Inc. 1994. Figures and Tables listed used with permission granted.
2. Bunnell, L. Roy. "Tempered Glass and Thermal Shock Index", ***National Educators' Workshop:Update 91 - Standard Experiments in Engineering Materials, Science and Technology.*** NASA CP-3151, 1992.
3. Kundu Nikhil K. "Graphing Techniques for Materials Laboratory Using Excel", ***National Educators' Workshop:Update 93 - Standard Experiments in Engineering Materials, Science and Technology.*** NASA CP-3259. April, 1994.
4. Kohne, Glenn S. and F. Xavier Spiegel. "An Automated Digital Data Collection and Analysis System for the Charpy Impact Tester", ***National Educators' Workshop:Update 93 - Standard Experiments in Engineering Materials, Science and Technology.*** NASA CP-3259. April, 1994.
5. McClelland, H.T. "Introduction to Usable Statistical Methods", ***National Educators' Workshop:Update 93 - Standard Experiments in Engineering Materials, Science and Technology.*** NASA CP-3259. April, 1994. April, 1994.

INCORPORATING "INTELLIGENT" MATERIALS INTO SCIENCE EDUCATION

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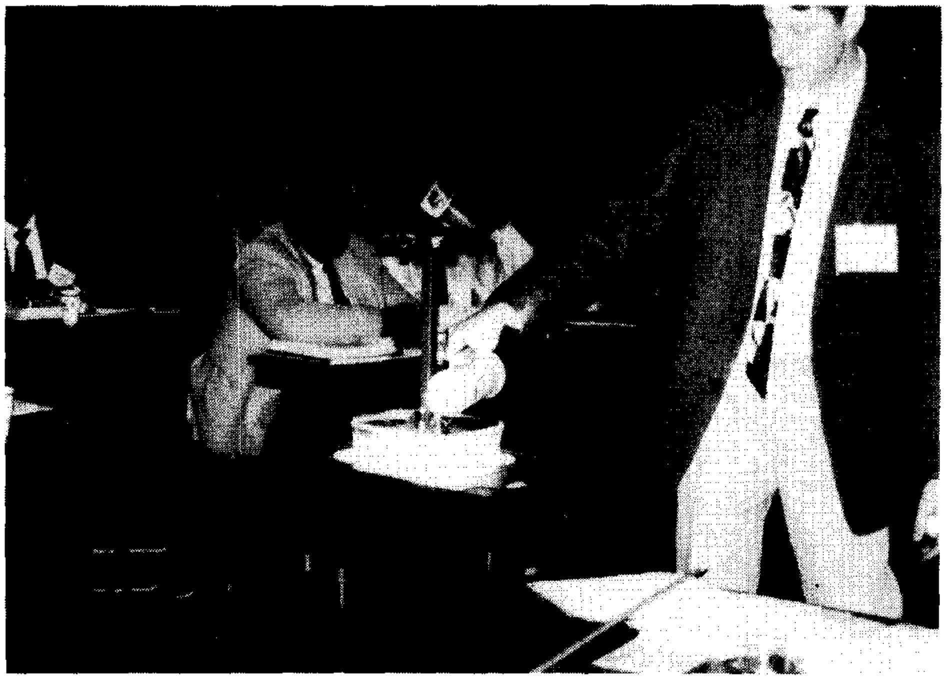

INCORPORATING "INTELLIGENT" MATERIALS INTO SCIENCE EDUCATION

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Presented at:

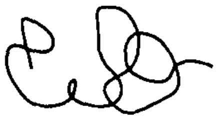
NEW/UPDATE 1994
Gaithersburg, Maryland
November 7, 1994

Outline

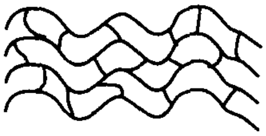
I. Rational and philosophy for the intelligent materials lab (8-10)
   A. Provide "real reality" in the classroom (8)
   B. Easily assimilated format
   C. Provide a lab where one does not exist due to cost or logistics (9,10)
   D. Free up instructor time for class preparation and student interaction
II. Review of basic concepts and themes covered in introductory polymer/materials class (1-7)
III. Handout of materials for exercises in polymeric and materials sciences.
   A. DOP plasticizer gradient of poly(vinyl chloride)
   B. Temperature responsive polymer, poly(N-isopropyl acrylamide)
   C. Ion concentration responsive polymer, sodium polyacrylate
VI. "Do the lab"

Basic Concepts and Themes in Polymer Science and Engineering (1-7)

Molecular Weight and Distribution



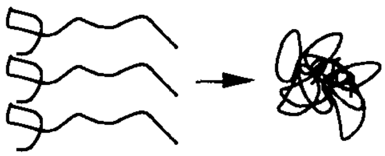
Thermoset/Thermoplastic



Steric Hindrances/Side Groups

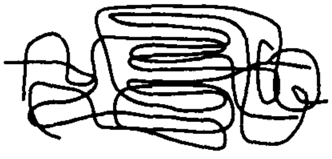
Polymerization

Intermolecular Forces/Cohesive Energy Density

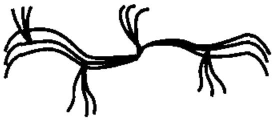


Entropy/Elasticity

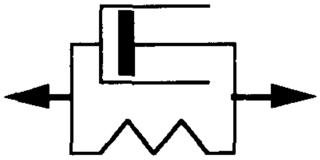
Morphology/Crystallinity



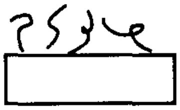
Temperature Transitions



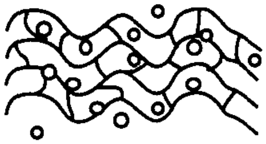
Viscoelasticity



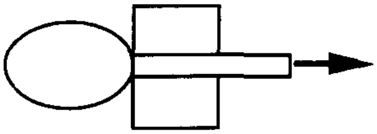
Surface versus Bulk Properties



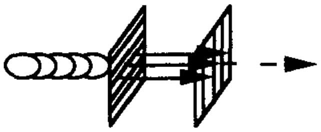
Modifiers/Additives



Processing/Fabrication



Optical Properties



Time-Temperature Superposition

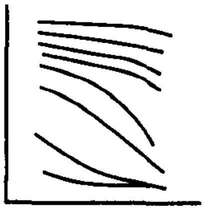


Table. Exploration material matrix matched to introductory polymer concept matrix.

Material System and Concept	Molecular Weight and Distribution	Morphology(Crystallinity)	Intermolecular Forces	Cohesive Energy Density/Solubility	Polymerization	Thermoset/Thermoplastic	Glass Transition	Time-Temp Superposition/Viscosity	Entropy	Steric Hindrance	Macromolecular Effects	Surface/Bulk Properties	Processing	Additives/Fillers	Optical Properties
Temperature Responsive Polymer				X						X	X				X
Ionic Responsive Polymer				X						X	X				X
Tg Homopolymers			X				X			X					
Heat Shrink Material		X	X					X		X			X		
Composites												X	X	X	
Surface Gradient												X			
Voight-Maxwell Models			X			X	X	X		X	X				
Shape Memory Polymer		X	X				X		X	X	X				
Crosslink Density Study	X		X			X					X		X		
Elastomer		X	X			X			X		X				
Polymerization Procedure	X				X					X	X		X	X	
Spherulite Gradient		X				X	X	X	X		X		X		X
Crosslink Gradient	X		X		X	X	X			X	X				
Plasticizer Gradient	X		X	X			X				X		X	X	
Copolymer gradient			X		X		X								

Rational and Philosophy

Most students come into polymer courses with various concepts and preconceptions which lead them to conclude that the behavior and properties of polymers are counter-intuitive. It is therefore important that they fully discover and observe the properties and behavior of polymeric materials for themselves.

A very large number of science and engineering courses taught in colleges and universities today do not involve laboratories. Although good instructors incorporate class demonstrations, hands-on homework, and various teaching aids, including computer simulations, the fact is that students in such courses often accept key concepts and experimental results without discovering them for themselves. The only partial solution to this problem has been increasing the use of class demonstrations and computer simulations.

We are developing a set of inexpensive and convenient hands-on discovery based experiments which the students can perform for themselves. The laboratory, although packaged like a textbook, will contain within it all of the materials, equipment, and information needed to directly discover and experience key concepts related to polymer materials.

The experiments are based on:

1) actual materials commonly utilized in consumer products, and
2) a set of specially developed materials which the students can utilize to directly see and experience complex concepts (8).

In both cases, the experiments and observations will utilize only the students' senses for transduction and detection.

Another important area of education which this research will impact is the growing field of distance learning. The Public Broadcasting Service (PBS) has developed a new program called "Going the Distance." In the PBS's definitive guide to distance learning, *Going the Distance* (9), major issues related to developing a distance learning program are discussed. The guide points out special needs of the laboratory sciences and distance learning. Others have addressed distance learning (9,10), and currently several states, including Utah, are strongly committed to the development of distance learning for secondary and post-secondary education.

Uniqueness: The difference between our proposed laboratory explorations and those of other, more common exercises is two-fold. The primary difference is in the required detection devices; our laboratories require the human perceptions of both sight and touch, others often require costly (though not more sophisticated) detection equipment. This is the very difference which allows the Labless Lab™ to be used outside the classroom. The secondary difference is the use of "intelligent materials" as teaching devices (8). The students can utilize theses materials to directly see complex polymer concepts.

Plasticizer Gradient (PVC)

Observation #1: Draping	Stiffness/Compliance Additives Low M.W. Components Molecular Weight Distributions Viscoelasticity
Observation #2: Indentation	Hardness Elasticity Viscoelasticity
Observation #3: Molding	Viscoelasticity Glass Transition Long Molecule Mobility Time–Temp. Relationship Processing Intermolecular Forces

** PVC is a very common commercial material (car seats, irrigation pipe, Tygon® tubing). Its wide range of uses and properties is due to different degrees of plasticization. This unique sample demonstrates this concept handily.

Shape Memory Polymer (polynorbornene)

Observation #1:

Comparison of mechanical properties at room temperature and at T>Tg

Glass Transition

Elasticity

Intermolecular Forces

Observation #2:

Mold polymer when T>Tg
and cool,
does it hold its shape?

Intermolecular Forces

Glass Transition

Observation #3:

Reheat above Tg

Molecular Mobility
Glass Transition
Intermolecular Forces

** This polymer can be purchased as a novelty from Maruyoshi® Co., Ltd. (Japan) Tel. 03(3470)6360. The raw/unformed polymer powder can be purchased from Zeon Chemicals, Inc. (USA) Tel. (708)437-9770. It has been proposed for use in medical devices, particularly arterial stents. The idea was to place the stent, in a collapsed geometry, into the artery; when the stent reaches body temperature it will expand to support the artery.

Surface Wettability Gradient (polystyrene)

Observation #1:

Sessile Drop Test

Intermolecular Forces §8.14, p.177

Surface and Bulk properties §17.20, p.425

Observation #2:

Peel Test

Intermolecular Forces §8.14, p.177

Surface and Bulk properties §17.20, p.425

Observation #3:

Capillarity Test

Intermolecular Forces §8.14, p.177

Surface and Bulk properties §17.20, p.425

** Polystyrene is used for food packaging and compact disc cases. It is well suited to these uses because of its transparency and hydrophobicity (it repels water). The transparency can be altered by adding a foaming agent which also increases its temperature insulation properties (Styrofoam™). The surface chemistry can be altered (oxygen or water plasma) to demonstrate the effect of chemistry on surface properties.

Temperature Responsive Polymer (poly(N,isopropylacrylamide))

Observation #1: Dry material properties compared to solution properties	Intermolecular Forces §8.14, p.177 Solubility and Concentration §8.14, p.177 Side Groups §15.6, p.327
Observation #2: Effect of different temperatures on solution properties	Intermolecular Forces §8.14, p.177 Solubility and C.E.D. §8.14, p.177 Optical properties §11.8, p.241 Side Groups §15.6, p.327
Observation #3: Reversibility – Heating and Cooling	Solubility and C.E.D. §8.14, p.177 Intermolecular Forces §8.14, p.177

** The primary use of this product is for controlled chemical delivery systems, where the polymer is used as a crosslinked gel. As a gel, the polymer expands and contracts in water solutions instead of dissolving and precipitating as did the nonlinked sample demonstrated. This is not a commercial polymer.

Ionic Responsive Polymer (sodium polyacrylate)

Observation #1:

Dry material properties
compared to
Hydrated gel material properties

Crosslinking §15.13, p.345

Elasticity §13.2

Morphology §11.8

Observation #2:

Affect of different solutions
(saline, sugar water, food coloring)

Intermolecular Forces §8.14, p.177

Solubility and Concentration §8.14, p.177

Side Groups §15.6, p.327

Observation #3:

Reversibility – Heat, Agitation

Molecular Mobility §12.1

Intermolecular Forces §8.14, p.177

Observation #4:

Solution Properties

Viscosity §9.13, p.207

Molecular Weight §9.2, p.187

Cohesive Energy Density §8.14, p.177

** Used in diapers to absorb liquids. Negative ions in solution take the place of the water and release the water. Can be purchased from Flinn Scientific Inc. Tel. (708)879-6900.

References

1. Cowie, J.M.G.: *Polymers: Chemistry and Physics of Modern Materials.* Chapman and Hall, 1991.

2. Billmeyer, F.W.: *Textbook of Polymer Science.* John Wiley and Sons, 1984.

3. Young, R.J.: *Introduction to Polymers. Chapman* and Hall, 1981.

4. Cambell, I.M.: *Introduction to Synthetic Polymers.* Oxford University Press, 1994.

5. Rodriguez, F.: *Principles of Polymer Systems.* McGraw-Hill, 1982.

6. Carraher, C.: *Polymer Chemistry.* Dekker, 1992.

7. Seymour, R.B.: Recommended ACS Syllabus for Introductory Courses in Polymer Chemistry. *J. Chem. Educ.*, vol. 59, 1982, p. 652.

8. Andrade, J.D.; and Scheer, R.J.: Applying 'Intelligent' Materials for Materials Education: The Labless Lab™. *Proceedings of the Second International Conference on Intelligent Materials.* C.A. Rogers, and Williams, G.G., eds. Technomic, 1994.

9. Toby Levine Communications, Inc. Bethesda, Maryland. *Going the Distance: A Handbook for Developing Distance Degree Programs Using Television Courses and Telecommunications Technologies.*" Annenberg/CPB Project and PBS Adult Learning Service, Corporation for Public Broadcasting and Public Broadcasting Service. 1994.

10. Watkins, B.T. "Uniting North Dakota." *The Chronicle of Higher Education.* Aug. 10, 1994, p. A17.

11. More details on preparation of these samples and their uses will be published in the *Journal of Materials Education* to be submitted mid-December.

MEASURING ENERGY LOSS BETWEEN COLLIDING METAL OBJECTS

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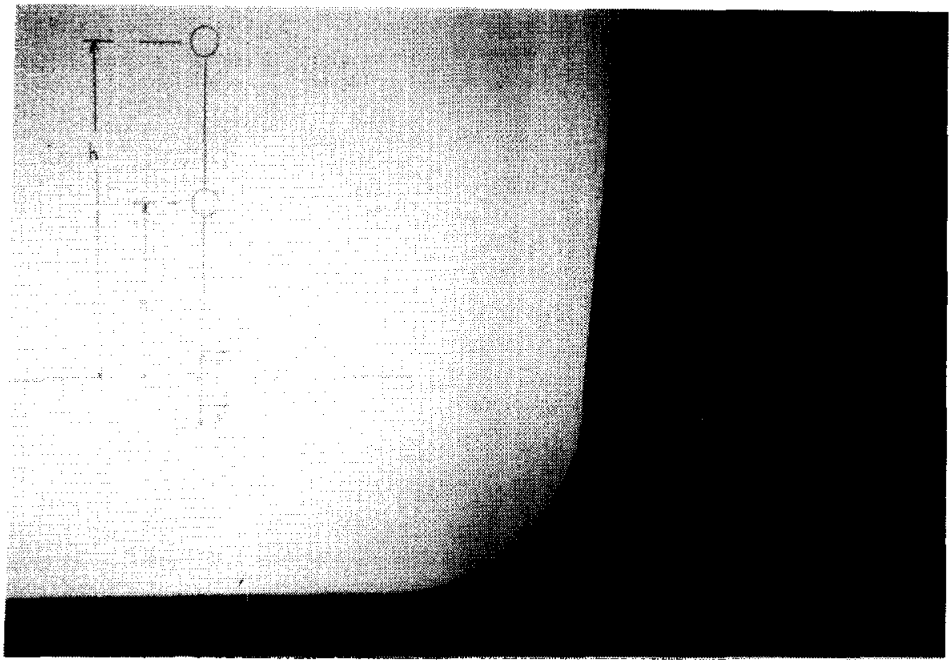
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Measuring Energy Loss between Colliding Metal Objects

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Key Words: Coefficient of restitution, kinetic energy

Prerequisite Knowledge: Knowledge of basic mechanics, conservation of energy

Objective: To determine the loss of kinetic energy when a metal sphere bounces off of a flat, hard metal surface. To determine how this loss of kinetic energy is related to the kinetic energy just before impact. To measure very short times electronically.

Equipment:
1. collection of metal spheres of various diameters
2. a small microphone
3. LM324 operational amplifier
4. 74121 monostable multivibrator
5. a "first and second pulse" detector
6. a 10 Khz crystal controlled square wave generator
7. a 7 digit counter
8. a meter stick accurate to 0.5 mm
9. a flat, hard, fairly massive metal surface
10. an RC - diode filter
11. 7414 schmitt trigger

Introduction

The coefficient of restitution between two colliding surfaces is defined as the ratio of the magnitude of the relative velocities after impact to the magnitude of the relative velocities before impact. If one object is falling and has a speed v just before hitting a stationary surface and rebounds with a speed v' just after hitting the surface, the coefficient of restitution[1] e can be defined as

$$e=\frac{v'}{v} \qquad (1)$$

In a perfectly elastic collision, e would be equal to 1. If the ball is dropped from rest from a height h, its kinetic energy just before hitting the floor is[2]

$$\frac{1}{2}mv^2=mgh \tag{2}$$

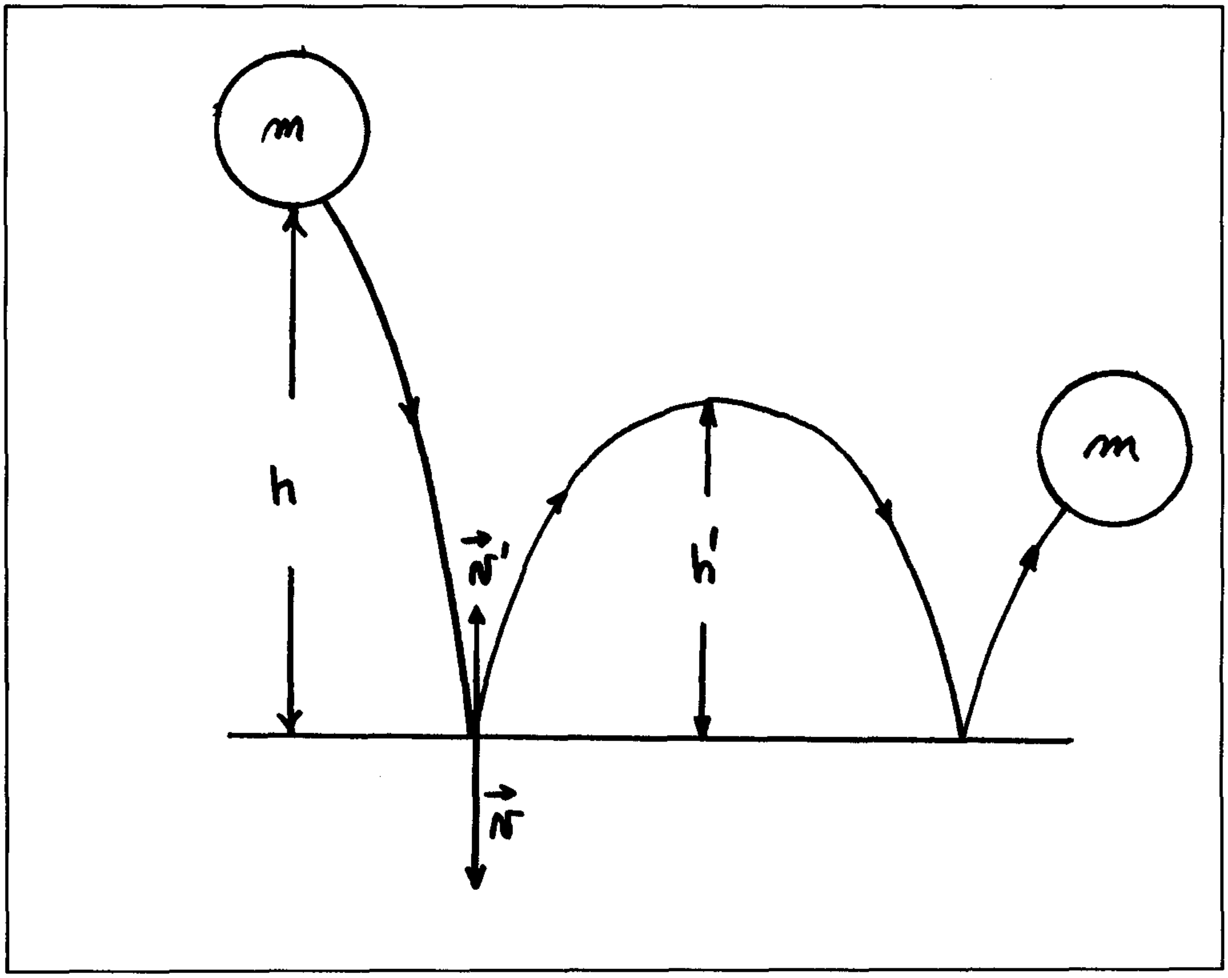


Figure 1 Falling Sphere apparatus

After the sphere rebounds, it will have a speed v' and will rise to a height h' where it will have potential energy

$$mgh'=\frac{1}{2}mv'^2 \tag{3}$$

Combining equations 2 and 3, we see that

$$(\frac{v'}{v})^2=\frac{h'}{h} \tag{4}$$

Thus from equation 1

$$e=\sqrt{\frac{h'}{h}} \tag{5}$$

Measuring h' becomes very difficult when h gets to be small. For this reason, an alternative approach has been developed to measure h' and thus e.

Since the sphere will rise to a height h' before coming to rest and starting to fall back to the flat surface, the time that it takes to come to rest at height h' is just t where[3]

$$h'=\frac{1}{2}gt^2 \tag{6}$$

from which we get

$$t=\sqrt{\frac{2h'}{g}} \tag{7}$$

The time for the sphere to hit the flat surface from the top of its trajectory is also t so the total time between the first and second bounce is T where

$$T=2t \tag{8}$$

Combining equations 5, 7 and 8, we get

$$T=e\sqrt{\frac{8h}{g}} \tag{9}$$

Thus for a given initial height h, we can measure the time T between bounces and find e.

$$e=T\sqrt{\frac{g}{8h}} \tag{10}$$

The quantity e is related to the loss in kinetic energy due to the collision. The kinetic energy before impact is given in eqn 2 while the kinetic energy after impact is given in eqn 3.
The loss of kinetic energy due to impact is

$$\delta KE=\frac{1}{2}mv^2-\frac{1}{2}mv'^2 \tag{11}$$

The ratio of the loss of kinetic energy to the initial kinetic energy is given by

$$\frac{\delta KE}{KE_0}=\frac{\frac{1}{2}mv^2-\frac{1}{2}mv'^2}{\frac{1}{2}mv^2}=1-\frac{v'^2}{v^2} \tag{12}$$

From equation 1:

$$\frac{\delta KE}{KE_0}=1-e^2 \tag{13}$$

And finally from equation 10:

$$\frac{\delta KE}{KE}=1-\frac{gT^2}{8h} \tag{14}$$

Thus for a given initial height h, the percentage loss of kinetic energy can be determined by the measuring the time between the first and second bounce.

Apparatus

In order to measure the times between bounces, we designed a circuit to detect the noise that the sphere made when it hit the flat surface. For this we used a microphone. After amplifying the output of the microphone, we fed the amplified signal to an analog comparator to produce outputs that were +5 volts or ground. These signals went to a schmitt trigger to sharpen the pulses and make them TTL compatible. Finally, a monostable multivibrator was triggered on the first 0 to 1 transition and stayed high for a time determined by its RC time constant until all the ringing from the noise vibrations were finished. The time was made long enough to mask all vibrations but the first and short enough to be ready for the next bounce. Effectively, each time the sphere bounced, a single logic 1 pulse was produced which stayed high for about 30 milliseconds. See Appendix I.

The output of this control circuit was fed to a pulse detector which was a digital circuit containing D type flip-flops which went high when the first pulse came along, low when the second pulse came along and stayed low for all following pulses until manually cleared. The output of this circuit was ANDed with a 10 Khz clock and the output of the AND gate went to a counter. Thus the number of counts was a direct measurement of the time T between bounces measured in tenths of milliseconds.

Procedure:

A metal sphere is held by a release mechanism which allows the sphere to be dropped from the same height on each trial. The distance h from the flat surface to the bottom of the sphere is measured using a meter stick and recorded in Table I. The counter is cleared and the "two bounce detector" is reset. The sphere is released. When the sphere hits the surface the first time, the counter begins to count. When the sphere hits the surface the second time, the counter stops counting. All subsequent bounces are ignored by the counter. The number on the counter is the time T between bounces in units of 0.0001 seconds. This number is recorded in Table I. This is repeated 5 times. The average and the standard deviation are computed and also entered into table I. This procedure is repeated for 7 different heights as indicated in Table I. We also measured and recorded the mass and the diameter of the sphere. The clock frequency is recorded in Table I as is the gravity constant g which appears in equation 14. In Table II, the percentage loss of kinetic energy is calculated from equation 14 using the experimentally determined values of h and T. The value of g is recorded as 9.80 m/sec^2. The coefficient of restitution e is also calculated using equation 10 and displayed in Table II.

Results:

The data in Table II is presented in two different graphs. In figure 2 the percentage loss of kinetic energy is plotted versus kinetic energy before impact and in figure 3 the coefficient of restitution is plotted versus initial height h. As the graphs indicate, the percentage of energy lost upon impact decreases with decreasing initial total energy and the coefficient of restitution e decreases with increasing initial height h. The student is given no explanation for this. He or she is asked to try to explain why this might be the case.

TABLE I . Data Collection for Sample Run							
h(cm)	1.00	2.00	3.00	4.00	5.00	6.00	7.00
run 1	503	668	785	894	960	1043	1099
run 2	504	663	780	876	959	1023	1105
run 3	497	660	778	871	957	1041	1112
run 4	499	660	777	885	944	1037	1109
run 5	498	668	783	887	954	1050	1100
Average	500.2	663.8	780.6	882.6	954.8	1038.8	1105.0
st.dev.	2.1	2.7	2.3	6.2	4.4	6.8	3.8
T(sec)	0.0500	0.0664	0.0781	0.0883	0.0955	0.1039	0.1105
mass of sphere=		0.012	kgm				
gravity g=		9.80	m/sec/sec				
diam of sphere=		1.43	cm				
clock freq=		10000	Hz				

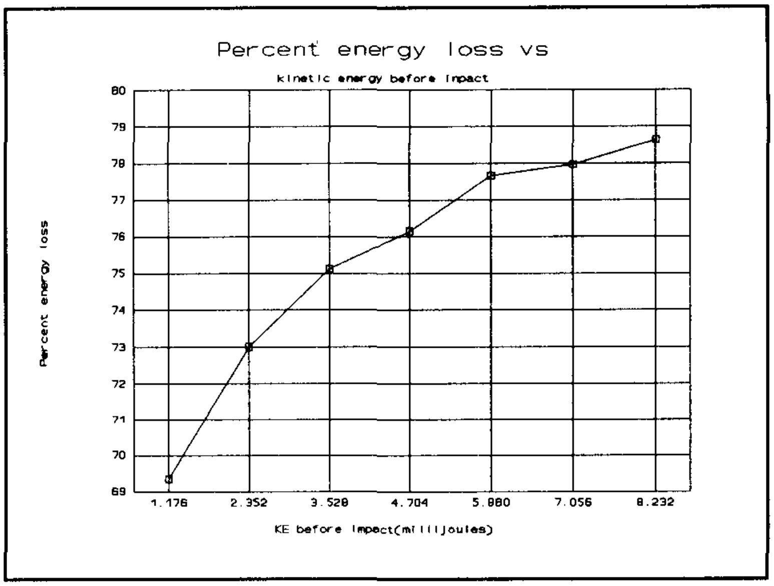


Figure 2 Percent KE loss vs initial energy

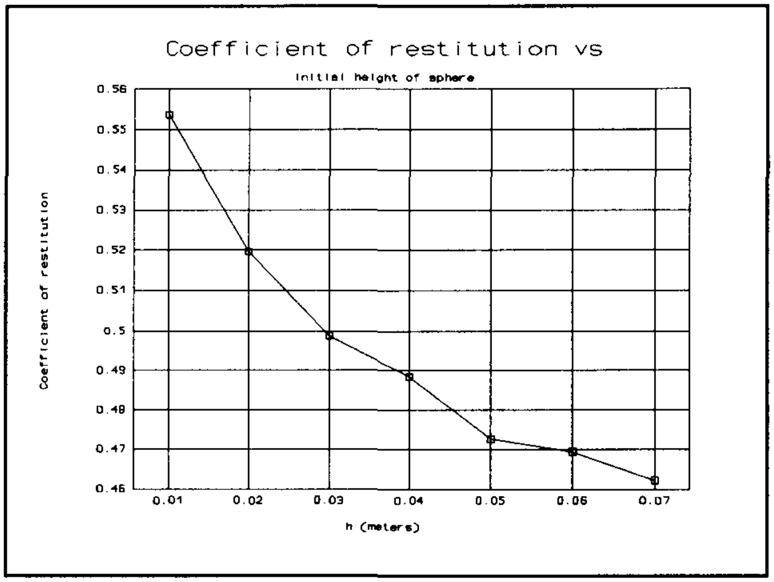


Figure 3 Coefficient of restitution vs initial h

TABLE II Calculations of e and energy loss

Avg	h(m)	Initial	% KE	e	
T (sec)		mgh(mJ)	loss		
0.0500	0.01	1.176	69.4	0.554	
0.0664	0.02	2.352	73.0	0.520	
0.0781	0.03	3.528	75.1	0.499	
0.0883	0.04	4.704	76.1	0.488	
0.0955	0.05	5.880	77.7	0.473	
0.1039	0.06	7.056	78.0	0.469	
0.1105	0.07	8.232	78.6	0.462	

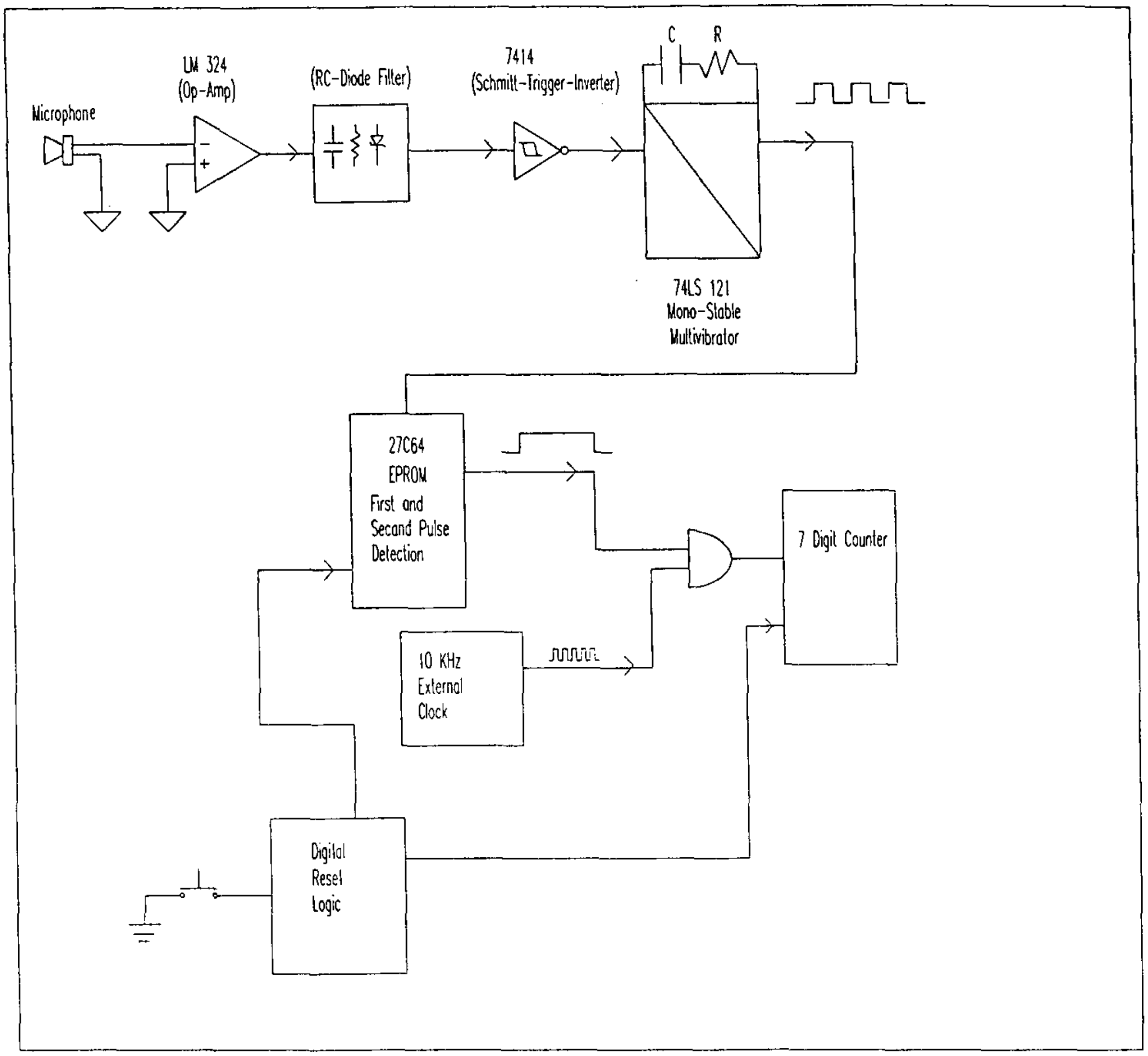
References:

1. Roller and Blum, *Physics Volume I,* Holden Day, p210

2. McLean and Nelson,*Engineering Mechanics - Schaum's Outline Series,* McGraw Hill, p320

3. R. C. Hibbeler, *Engineering Mechanics: Dynamics,* Macmillan, p439

APPENDIX I BOUNCE DETECTION CIRCUIT



STRESS CONCENTRATION: COMPUTER FINITE ELEMENT ANALYSIS VS. PHOTOELASTICITY

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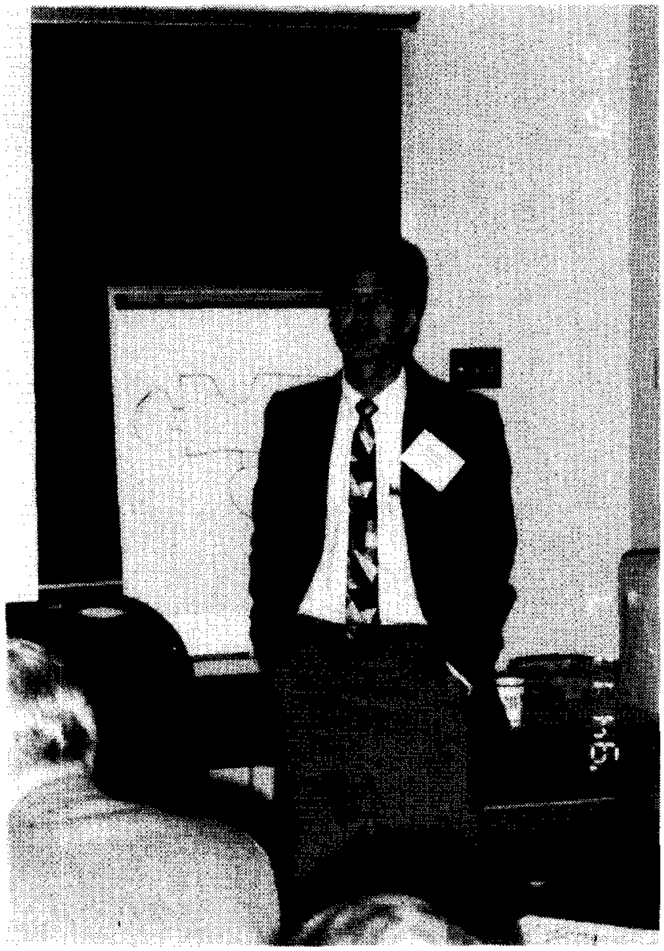

Stress Concentration: Computer Finite Element Analysis vs. Photoelasticity

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Key Words: finite element analysis, stress concentration, photoelasticity, maximum stress

Prerequisite Knowledge: Students should have a basic understanding of applied strength of materials through principal stresses and strains, and a prior laboratory covering photoelastic methods is recommended. Students should be computer literate. Prior computer aided design training is helpful when developing the geometry of the computer models, but is not required.

Objective: To gain insight into stress concentration due to geometric discontinuities. To compare computer finite element analysis with traditional methods used to determine stress concentration factors and maximum stress at geometric discontinuities.

Equipment:
1. Computer with finite element analysis software
2. Birefringent plastic test specimen(s)
3. Transmission polariscope with load frame and load cell
4. Digital readout for load cell
5. Tables of stress concentration factors for various geometric discontinuities and loadings

Introduction: The elementary formulas for average stress and strain presented in most mechanics of materials courses are based on members that have constant, or gradually changing, cross-sectional area. However, this is not usually the case in actual machine parts or structural members where shoulders, notches holes or other geometric discontinuities result in a modification of the simple average stress distribution. A small discontinuity in a member will generally increase stress locally, with the increase being out of proportion when considering the nominal cross-sectional area and applied load. The stress raising effect of a discontinuity results in a maximum stress that normally occurs at the boundary of the discontinuity. These maximum stress values must then be address when designing actual load bearing members.

The stress concentration factor (SCF) is the ratio between the maximum stress and the nominal stress. Values for the SCF for members with common geometric discontinuities (notches, holes, shoulders, etc.) can be found in tabular form (1,2) for various load cases. Multiplying the SCF by the nominal stress returns the maximum stress in the cross-section containing the discontinuity.

The photoelastic method can also be used to determine maximum stress at a discontinuity, and provides a visual representation of the stress distribution in the model. When using a transmission polariscope, a birefringent, uniform cross-section calibration specimen is used to determine the fringe constant of the modeling material. SCF and maximum stress can then be calculated using the photoelastic stress optic formula (3). Care must be taken to obtain a number of readings when using photoelasticity, since the load at which a fringe becomes visible is an arbitrary decision made by the observer.

Microcomputer based finite element analysis (FEA) also provides a visual image of the stress distribution around a discontinuity, and the FEA program will provide values for stress at any location on the computer model. Comparing the computer generated image with that found from the photoelasticity test is a quick way to verify the accuracy of the computer model. Experimental verification of FEA models helps students gain confidence in their ability to correctly develop the computer models. The computer modeling was done using the microcomputer version of COSMOS/M, Version 1.61, from Structural Research and Analysis Corporation.

Procedure: The maximum stress at a discontinuity is to be found and compared using three different methods; computer FEA, reference tables and photoelasticity. The pattern of stress distribution developed from FEA will also be compared to that observed using photoelasticity.

The test specimen is made of a birefringent plastic that is 0.117 inches thick, and has a modulus of elasticity of 360,000 psi (see Figure 1). The load is to be 50 pounds tension along the x-axis, applied at the ends of the model or test specimen. It is recommended that the experiment proceed in the following order: FEA, reference tables, photoelasticity.

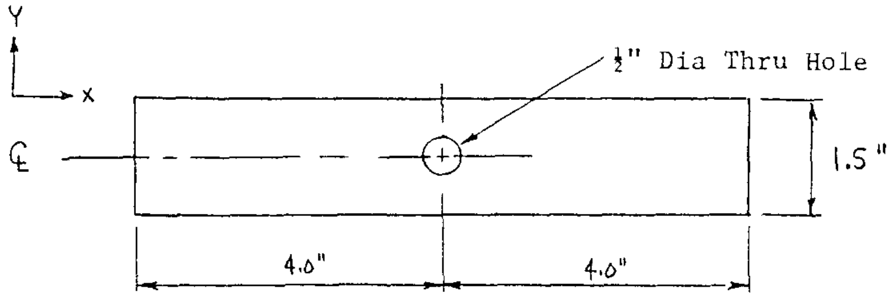


Figure 1. Model used for stress concentration experiment

Finite element Analysis (FEA): Regardless of the object to be modeled, finite element analysis follows the same basic pattern; the user must specify model geometry, element type, mesh densities, section properties, material properties, constraints and loading. After the model specifications are complete, the type(s) of analysis required is executed and then the output form is specified. This output can then be compared to that found using the reference tables or photoelasticity.

First, the model geometry is input per the specifications in Figure 1. This can be quickly accomplished since the model consists of just two basic geometric shapes, a circle and a rectangle, and will be represented as a two dimensional model with constant thickness.

The stress concentration specimen can best be represented by two dimensional elements that have a constant thickness. COSMOS/M uses the term PLANE2D to represent this type of element. The surface of the computer model must now be "meshed", which means that the surface of the model is to be completely covered with PLANE2D elements that are joined at their corners, or nodes.

Typically, high mesh densities (many elements in a unit of area) will return more accurate results than low mesh densities since values for stress and strain will be computed for each element. However, increasing the number of elements increases the computer time required to run the FEA model, so high mesh densities should only be used in areas of specific interest, such as the area around the discontinuity. The stress concentration model in Figure 1 is also symmetric about both the X and Y axes, so this specimen can be most efficiently modelled through the use of a quarter section to further reduce the problem size. Boundary conditions must now be enforced on the quarter section so that it properly represents the entire stress concentration specimen (4).

The 50 pound tensile load is then applied to the computer model, and a static analysis is specified. Results from the analysis can then be displayed on the computer screen as a stress plot, as shown in Figure 2. The stress distribution from the computer model can now be visually compared with that of the photoelasticity experiment. Values for the magnitudes of the stress contours are also shown on this plot.

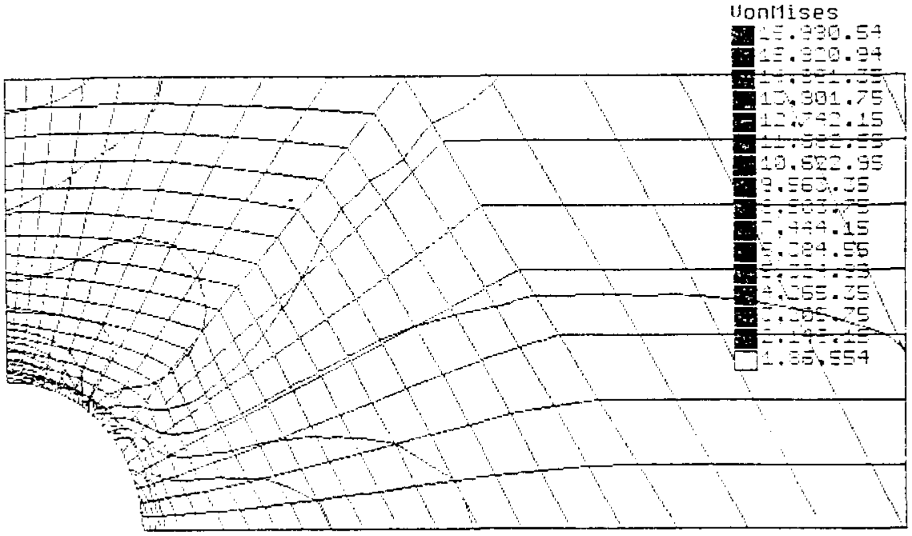
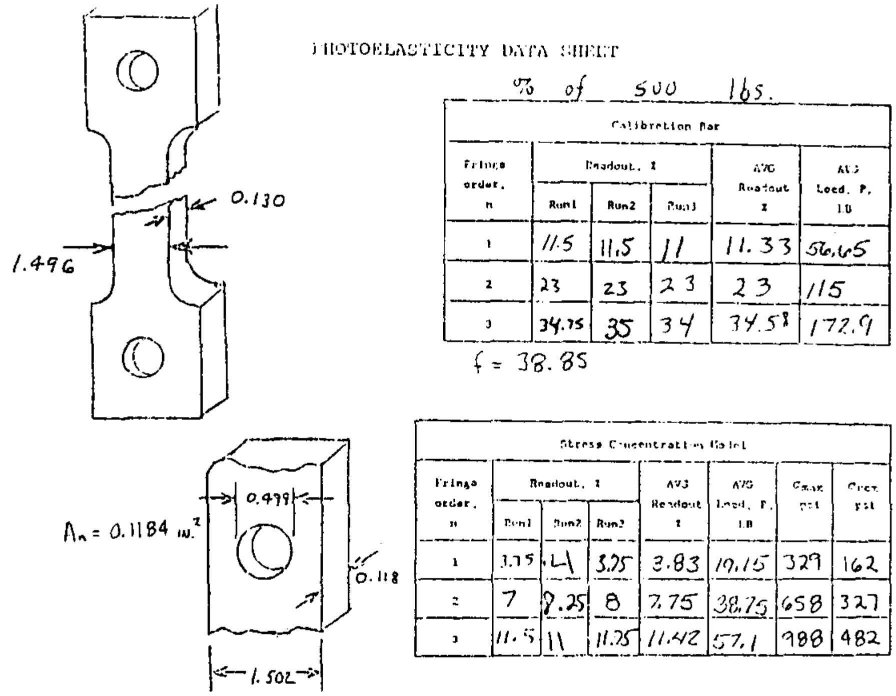


Figure 2 Stress distribution plot

Reference tables: Students use reference tables (1,2) to determine the stress concentration factor (SCF) for a model with geometry as shown in Figure 1. The nominal stress is then calculated based on the cross-section that contains the discontinuity. The maximum stress is found from multiplying the nominal stress by the SCF. This can then be compared to the results for maximum stress found using the FEA model.

Photoelasticity: Students are given the stress concentration data from a previous Applied Strength of Materials laboratory, as shown in Figure 3. The data include the specimen dimensions, fringe constant for the specimen material, and sufficient information from the experiment so that a stress concentration factor can be calculated. The maximum stress due to a 50 lb. applied load is calculated, and can then be compared with results based on both reference tables and FEA.

A birefringent test specimen (see Fig. 1) is then placed in the load frame of the polariscope and a 50 lb. load is applied. The stress distribution pattern developed is observed and compared to the stress distribution developed using finite element analysis.



PHOTOELASTICITY DATA SHEET

% of 500 lbs.

Calibration Bar

Fringe order, n	Readout, %			AVG Readout %	AVG Load, P, LB
	Run1	Run2	Run3		
1	11.5	11.5	11	11.33	56.65
2	23	23	23	23	115
3	34.75	35	34	34.58	172.9

f = 38.85

Stress Concentration Model

Fringe order, n	Readout, %			AVG Readout %	AVG Load, P, LB	σ_{max} psi	σ_{nom} psi
	Run1	Run2	Run3				
1	3.75	4	3.75	3.83	19.15	329	162
2	7	8.25	8	7.75	38.75	658	327
3	11.5	11	11.75	11.42	57.1	988	482

Figure 3 Photoelasticity data sheet

Conclusions: A table of the maximum stresses found using each method should be developed for comparison of the results. A comparison of the stress distribution patterns found by FEA and photoelasticity can be done using photographs of each technique, however it may be more convenient to set up a microcomputer next to the polariscope and allow students to plot their FEA results during the photoelasticity experiment for a real-time comparison.

Instructor Notes: This experiment was used in a course on Applied Finite Element Analysis as an exercise in experimental verification of FEA results. Students with a solid CAD background and good computer skills are able to quickly learn to develop FEA models, but seem to have difficulty with properly meshing a model, applying boundary conditions and properly interpreting results. The use of proper mesh densities is easily shown using a very simple model that includes a stress concentration; higher mesh densities near the discontinuity for greater accuracy, lower mesh densities in the main body of the model for efficiency. The model used here also provides an opportunity to develop boundary conditions for the quarter section used in the FEA analysis. Students have a tendency to accept any output generated by an FEA program, so the verification experiments are used as a tool to increase students' confidence in their ability to generate accurate FEA models.

References:

1. Roark, Raymond J., Roark's Formulas for Stress and Strain, 6th Edition, McGraw-Hill, Inc., New York, NY 1989
2. Cheng, Fa-Hwa, Applied Strength of Materials, Macmillan Publishing Company, New York, NY 1986
3. "Student Manual on the Photoelastic Coating Technique", Bulletin 315-A, Measurements Group, Inc. 1984
4. Baran, Nicholas M., Finite Element Analysis on Microcomputers, McGraw-Hill Book Company, New York, NY 1988

FAILURE ANALYSIS OF INJECTION MOLDED PLASTIC ENGINEERED PARTS

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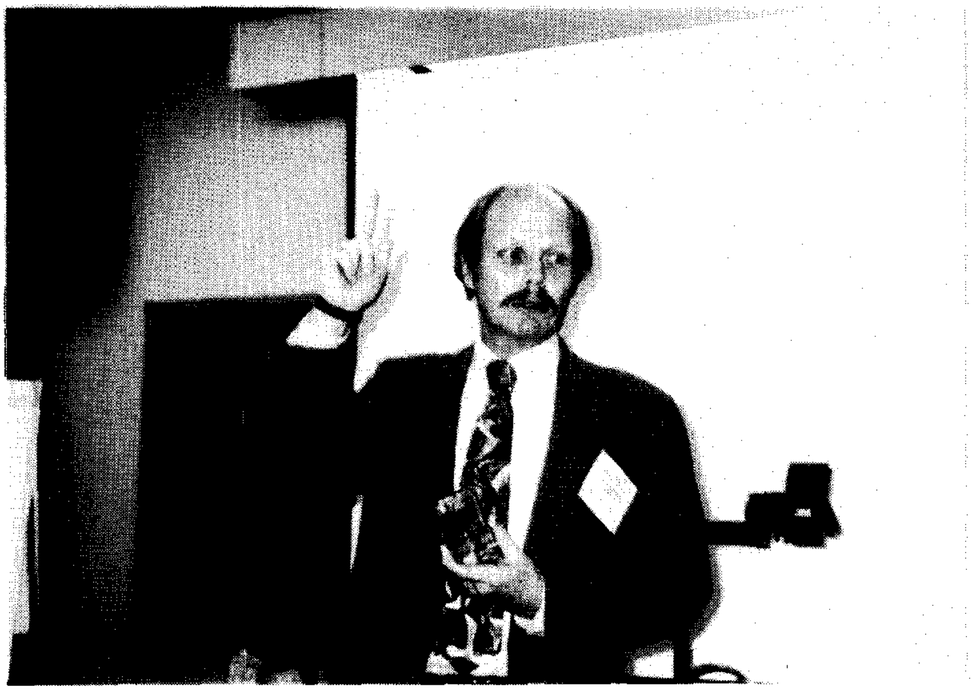

Failure Analysis of Injection Molded Plastic Engineered Parts

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Key Words: Fractography, Fracture, Service life, Polymer Degradation, Failure Analysis,

Prerequisite Knowledge: Knowledge of plastics injection molding, materials testing, quality control, plastics as an engineered material, and ASTM standards are beneficial.

Objective: To locate a suitable injection molded engineered part. Analyze the part, materials selection, probable processing characteristics and design as dictated by the parts intended function, and develop a plan using design of experiments (DOE) to exhibit planned laboratory failure.

Equipment: Various test equipment sensitive enough to show results when testing plastics, i.e., impact, tensile, compression, friction, hardness, stiffness, dimensional, melting point and viscosity/flow characteristics, plus other tests as needed.

Various chemicals, fixtures, gages, environmental conditions, ovens, analytical balance, software packages (statistical, material selection, part design, and other packages) to assist in the analysis of the parts.

Multiple copies (approximately 6) of the selected part to be tested and an abundance of insight, and analytical, synthesis and observation skills plus the time required to thoroughly analyze the implications of the DOE process on part failure.

Introduction:

Failure as defined by ASTM D 1780, (one of many related definitions) is rupture of the specimen, or exceeding the strain requirements of a specific design. Whereas analysis is defined by ASTM E 135 as the ascertainment of the identity or concentration, or both, of the constituents or components of a sample. Failure analysis can then be defined as the ascertainment of the identity of the strain(s) exceeding the requirements of a specific design.

This laboratory combines failure analysis with design of experiment, it is to be used in a senior level DOE (ITMFG 425) capstone course. The laboratory is intended to last for 8 to 10 weeks and represents a major portion of the lab grade. Design of experiment is defined as the process of planning an experiment. Failure analysis and DOE is quite literally a planned experiment to evaluate the design of a product after the fact. Designing an experiment can work both in the assembly of a product (manufacturing) and also in the disassembly, gradual or rapid failure of a product or component. The benefit of using DOE in failure analysis gives one a target, the failure of a part or product. Working backwards from the failure one can produce a more robust product. What is involved in this process can range from but is not limited to the topic of DOE to systems design, materials testing, testing standards, material, processing, applications of technology, literature searches, materials testing, processing variation and control, software, data collection, and fixtures and fixture design (gage R&R).

Failure analysis of an injection molded plastic product is a convenient, but not an easy approach to understanding the impact of DOE, materials and processing. The plastic products are readily available, selection of the part itself requires some engineering analysis, the cost is minimal, the methods used to induce failure are varied, and fixturing and testing of the parts can be done quite easily. The problem with using injection molded parts may be the quantity of unknown items, which is at times typical in failure analysis.

A cornucopia of problems associated with the actual part failure exist. The researcher must find a plausible, defensible cause for failure. With an injection molded part causes for failures (to list a few) may be any one or a combination of the following;

Materials problems
    Thermal degradation
    Excessive molecular weight distribution
    Appropriate viscosity or changes in viscosity
    Sampling procedures
    Material selection
    Blends of material
    Contaminants
    etc...

Part design
    Proper radii
    Size of bosses
    Fasteners and their use
    Forces anticipated
    Cost
    Location of sprue, runners and gates
    etc...

Processing
    Material conditioning
    Resident time
    Internal pressures and shear characteristics of material
    Internal temperatures
    etc...

Consumer use and abuse
    Chemicals
    Temperature
    Radiation
    etc...

Several of the experimental stress analysis techniques that have been developed for metal applications can be used for injection molded plastic parts. Some of the experimental techniques used for stress analysis are:

- Brittle coatings
- Strain gages
- Photoelasticity
- Solvent testing.

Brittle coatings are based on perfect adhesion of a strain sensitive coating applied on the surface of the part to be tested. As the part is loaded, strains developed in the part are transmitted to the coating which first begins to crack at its sensitivity threshold in the area of the largest principal stress. The cracks tend to be perpendicular to the direction of the principal stress.

Strain gages of several types exist, acoustical, electrical, mechanical, optical and pneumatic. Electrical strain gages tend to be the most readily available and can be very easily adapted for use in failure analysis. There are four major categories of electrical resistance strain gages:

1. Unbonded-wire gages
2. Bonded-wire gages
3. Bonded-foil gages
4. Piezoelectric gages.

The most popular and useful for molded plastic part use is the bonded-foil gages. Foil gages can be cemented easily to almost any shape. The co-efficient of linear thermal expansion of plastics is a factor to consider when using sensitive strain gages.

Photoelasticity - Using polarized filters and white light with some transparent and opaque plastics (ASTM D 4093), they will tend to show fringe patterns. The fringe patterns represent the overall stress distribution present in the part. Interpretation of the fringe patterns can lead to quantitative measurements of the magnitude and direction of the stress as molded into the part or induced through handling or the part in its actual environment.

Solvent testing - Stress as molded into a part or applied has an adverse impact on the chemical resistance of the material. The molded part can be saturated (sprayed or dipped) with a solvent of predetermined strength at a specified time and temperature, then cleaned to observe cracks and/or crazing. Any visual changes indicate that the stress levels in the area of the molded part are greater than those not effected. This method is indicative of stresses only, either that the stresses (compressive or tensile) are present or absent. Typical household solvents (cleaners) can be used with a variety of materials, the solvents are very specific to a particular plastic. Toluene with n-propyl alcohol and ethyl acetate with methyl alcohol works well with polycarbonate relative to generating cracks or crazes with reference to stress concentrations.

Depending on the part design, material selection, and application various combinations of the above techniques can be used with typical equipment and fixtures available in a materials test laboratory and/or metrology laboratory. For example, photoelastic measurements can be used in combination with a coordinate measuring machine. Fringe patterns/order and associated stresses on the part can be correlated over a range of forces on the part. Solvent testing can also be incorporated to enhance failure and analysis.

Procedure:

1. Obtain an injection molded engineered plastic part, depending on the DOE, obtain approximately six duplicate parts. Part selection should be based on the intent of this laboratory, DOE/failure analysis, not on availability of parts.

   Many products exist that are suitable for this laboratory. The list of products are endless and can be found in stores ranging from industrial supply (hydraulic, electrical, plumbing, and construction) to office, medical, engineering supply and even housewares.

   Collect and document all available information relative to the part, where it will be used, what properties might be expected of the part, what will be the expected environmental conditions for the life of the part (this includes shipping), consider consumer use and abuse, processing considerations and any other supportive information relative to your failure analysis.

   The following may provide some insight into the selection criteria for the part;

   Design characteristics of part
     Mating part characteristics if any
       Bolts-torque settings and variation
       Screws-design and size variation
       Snap fits
       Bonding
       Drilled holes

            - Molded in holes
            - Part loading

    - Gating of part and system
        - Plastic flow into cavity
            - Amorphous polymer chain orientation
            - Crystalline polymer chain orientation
            - Sprue/runner/gates
                - Size
                - Location
                - Balanced cavities

    - Polymer materials properties
        - Physical
        - Mechanical
        - Dimensional
        - Chemical
        - Electrical
        - Processing

    - Part environment
        - Temperature
        - Humidity
        - Light
        - Chemicals
        - Pressure/vacuum
        - Stress concentrations

- What properties can be tested in your existing facilities?
    - Mechanical
    - Physical
    - Dimensional
    - Chemical
    - Electrical

2. Assume failure(s) of a particular type, defend and justify the parts possible failure(s) based on;
    - Literature search
        - Material characteristics
        - Processing precautions-sensitivity to heat/moisture
    - Design criteria of specific material
    - Material selection
    - Processing characteristics
        - Gating
        - Amorphous/crystalline
        - Processing data
    - Material handling
        - Drying
        - Assembly processes
            - Machining
            - Bonding/joining/etc...

3. Develop the DOE to induce laboratory failure and maintain subsequent analysis and documentation.

Determine possible failure analysis schemes
- Develop quantitative analysis procedures to support findings, know what properties need to be evaluated and how they will be evaluated.
- Consider fixtures needed for testing
- Consider testing options
- Identify critical properties

Develop test criteria to show slow gradual part failure (planned vs. actual) using available equipment as listed. Chemical stress analysis may be used as needed to enhance failure. A clear or transparent part will facilitate the use of ASTM D 4093 and make failure analysis more thorough.

Slow failure implies gradual failure as evidenced by a variety of quantitative changes over time, i.e., visual, dimensional, shape, etc...

Develop quantitative analysis procedures to support findings. Document what works vs. what doesn't work to highlight tendency towards part failure. Maintain an accurate log of all activities.
- Select the appropriate part tests based on available;
  - Equipment
  - Environment
  - Tools
  - Fixtures (fixture design)
  - Fixture availability
  - Sampling procedure
    - Samples available
    - Materials ID
    - Material specs
  - Time to run tests
  - Quantitative data available (data collection)
  - Forces used vs. time required for results

Notes to the Instructor:

Keep the selection of parts to the less complicated designs.

Keep testing and fixtures as simple as possible.
- Rubber tooling
- Epoxy
- Other cast materials

Assume sample sizes of 1 or 2 are acceptable.

Assume data from test results using a small sample size are valid.

Because of the possible complexity of this project, encourage students to work in groups of 2 to 3.

References:

A.J. Durelle, E. Phillips and C.H. Tsao, Introduction to Theoretical and Experimental Stress Analysis, McGraw-Hill Book Company, NY, 1958.

J.W. Dally and W.F. Riley, Experimental Stress Analysis, McGraw-Hill Book Company, NY, 1965.

R.D. Moen, T.W. Nolan and L.P. Provost, Improving Quality Through Planned Experimentation, McGraw-Hill Book Company, NY, 1991.

A.S. Redner and W.E. Nickola, Measurement of Residual Strains and Stresses in Transparent Materials, Experimental Techniques, Vol. #2, February 1984.

S.R. Schmmidt and R.G. Launsby, Understanding Industrial Designed Experiments, Air Academy Press, Colorado, 1992.

DEMONSTRATIONS IN MATERIALS SCIENCE FROM THE CANDY SHOP

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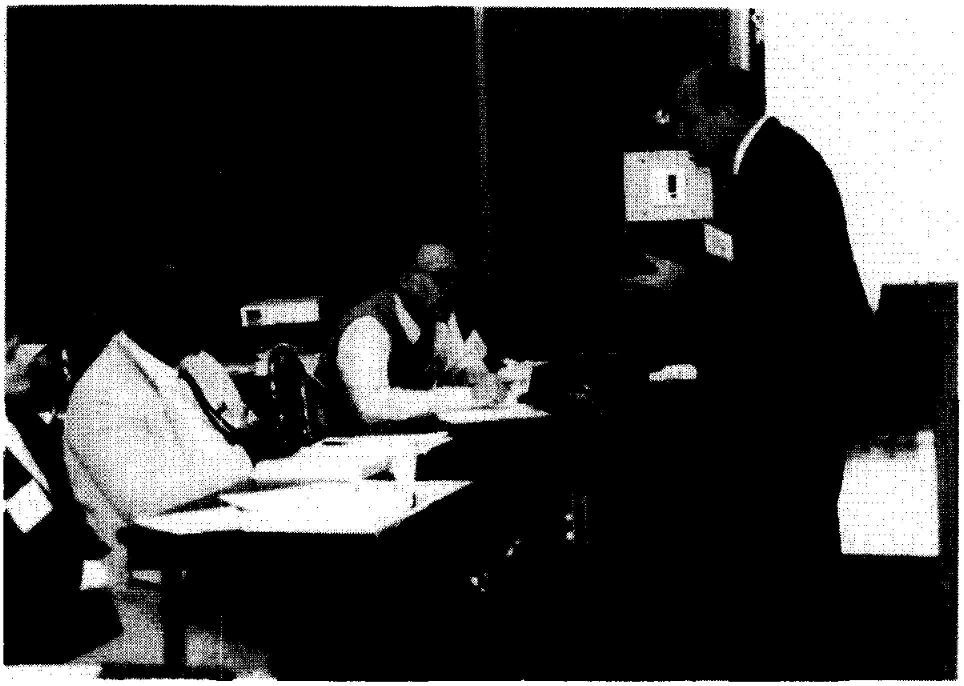

DEMONSTRATIONS IN MATERIALS SCIENCE FROM THE CANDY SHOP

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ABSTRACT; Readily available items from the candy counter at any supermarket or other stores can be used to demonstrate such properties as elasticity, plasticity, composite behavior, ductile and brittle fractures and luminescence.

REQUISITE KNOWLEDGE; Curiosity.

KEY WORDS; Composite, Elasticity, Plasticity, Ductility, Fracture, Triboluminescence.

OBJECTIVES; The purpose of these demonstrations is to illustrate some interesting properties of some candy products. [1,2]

EQUIPMENT AND SUPPLIES;

1) Chewing gum (sticks)
2) Chewing gum (coated)
3) Hard candy
4) Coated chocolate candy
5) Taffy
6) Peanut brittle
7) Licorice
8) Wintergreen mints

CHEWING GUM; Pull a stick of gum from both ends. The gum will deform in a ductile manner, i.e. when it fractures, you can note a necking area and considerable deformation. If the gum is put in the freezer to cool for about fifteen minutes, you can cause the gum to break in a brittle manner. This phenomenon is known as a ductile to brittle transition, and is very important in designing structures which will experience variations in temperature. A similar fracture can be demonstrated if you allow the gum to lose moisture and become stale. The loss of moisture causes the gum to become less ductile. Wood exhibits a similar behavior and has drastically different mechanical and thermal properties when the moisture has been removed. Taffy can be substituted for gum in this demonstration.

COATED GUM; Gum is coated for several reasons such as to give the customer a double treat, a sweet rather hard candy and chewing treat, and also for convenience of packaging and the added convenience of not needing a wrapping. Coated gum is an excellent example of one type of composite where a coating is placed on another material to protect the inner material from deterioration. Regular gum without a wrapper or coating would become sticky and lose its appeal, and usually not be very appealing. Coatings such as zinc on steel, paint on wood or walls, and asphalt on foundations are used to protect these materials from the outside elements and provide corrosion resistance, esthetic appeal, and waterproofing respectifully. Coated chocolate candy can be used in this demonstration.

HARD CANDY; Any hard candy can be used to demonstate a brittle fracture. Hard candies are meant to last longer than other candies and give the consumer a lengthier taste experience. Striking a hard candy lightly with a hammer will result in several pieces of candy. Quite often designers need very hard materials which can withstand large loads without deformation. Foundations and superstructures for large buildings are examples of brittle materials.

PEANUT BRITTLE; Peanut brittle is a combination of a syrup (taffy-like) and nuts. The nuts are added to the syrup to provide a double treat, and this addition of nuts also changes the mechanical properties. The pure brittle would act in a typical brittle or hard manner, however when the nuts are added the brittle is strengthened. Although this may not mean much to the consumer it is an important materials result. This phenomenon is an example of another type of composite. In this case two different materials are combined as a mixture to enhance the properties. In the case of peanut brittle,the manufacturer wants to enhance the taste, and incidentally has changed the mechanical properties. Concrete is a mixture of stones and cement which is the most abundant building material in the world. The Egyptians added straw to clay to make more durable bricks, and steel or composite bars or mesh are added to concrete to improve the load bearing capacity.

LICORICE; Licorice sticks come in a variety of colors. The color is added to appeal to the desires of the consumer. If a licorice stick is pulled gently from opposite ends and then released the licorice will deform, but then almost immediately return to its original length, pull a little harder and it will deform but take a little longer to return to its original length, pull even harder and the licorice will become permanently deformed. This behavior is known as elasticity, anelasticity and plasticity. Continued pulling on the licorice will cause a ductile fracture. Many engineering materials are designed to take advantage of this range of behavior of materials. Certain alloying elements are added to a material to change the range or limit of this type of behavior. Carbon in iron, silicon in aluminum, copper in zinc and tin in copper are excellent examples.

WINTERGREEN MINTS; Any wintergreen mint has the wintergreen flavor added to enhance the taste and appeal to a certain consumer. However, the wintergreen flavor enhancer adds an unexpected added attraction. When the mint is broken in a darkened room, you will note a flash of light. This phenonmenon is called triboluminescence. A mechanical deformation results in the emission of light energy. Other examples are bioluminescence, chemoluminescence and electroluminescence, each named for the phenomenon that causes the emission of light. Examples include fire-flies, a propeller's wake at night, a swimmer's wake at night, certain xhemical reactions, etc.

CONCLUSIONS; The author has performed these experiments and demonstrations to audiences of all ages with enthusiastic results. [3,4,5] Quantitative results can be measured and each demonstration can be expanded and/or altered to your desires. You are only limited by your imagination and curiosity.

REFERENCES;

1. Jacobs, James A. and Kilduff, Thomas F. : Engineering Materials Technology, Prentice-Hall 1993.

2. Fellers, William O.: Materials Science, Testing and Properties for Technicians, Prentice-Hall 1990.

3. Spiegel, F. Xavier: Five experiments in Materials Science for less than $10.00, NASA Conference Publication 3151 November 1991 263-265.

4. Spiegel, F. Xavier: Experiments in Materials Science from Household Items, NASA Conference Publication 3201 November 1992 373-375.

5. Spiegel, F. Xavier: Inexpensive Materials Science Demonstrations, NASA Conference Publication 3259 November 1993 119-124.

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OPTIMIZING WING DESIGN BY USING A PIEZOELECTRIC POLYMER

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AN AFFORDABLE MATERIAL TESTING DEVICE

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Optimizing Wing Design by Using a Piezoelectric Polymer

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Sponsoring Teacher at John Adams High School: Nevin E. Longenecker

Objective:

This experiment allows students to learn how to use electronics with an electromechanical polymer to monitor wing stability. By using this polymer, changes in air pressure around a model wing can be detected and used to determine stable wing designs.

Equipment:

- LM741 or LM1458 Operational Amplifiers
- CHAMP digitizer interface
- Apple IIe computer
- Wind Tunnel

Introduction:

The principle of airplane lift is based in the design of its wings. Thrust is necessary to produce an air flow around the vehicle so that lift is achieved. The amount of thrust required to lift the plane into the air is dependent on the wing design and the mass of the vehicle. The lift that an airplane produces is determined by the difference in air pressure on the wings' lower and upper sides. This pressure difference occurs because the velocities of the air on the top and the bottom of the wing are different.

The relationship between air velocity and air pressure was first discovered by Daniel Bernoulli. Bernoulli observed that as the velocity of air increased the air pressure decreased and vice versa. Since the air velocity above the wing is higher than the air velocity below the wing, the air pressure is lower and higher respectively. If laminar air flow (as opposed to turbulent air flow) exists, then this principle can be applied and lift is produced.

In this experiment, the piezoelectric polymer polyvinylidene fluoride (PVDF) was used to measure stability in several wing designs. Piezoelectric materials have the ability to convert a mechanical force into an electrical pulse and vice versa. The molecules in this polymer are arranged in a zig zag formation where two fluorine atoms and a carbon atom form a dipole which causes a charge redistribution. Likewise two hydrogen atoms are bonded to a carbon atom which causes an opposite charge redistribution (fig 1). When the film is exposed to a strong DC field, hydrogen and fluorine molecules align as dipoles. This causes an electric field polarization to exist within the film. When the dipoles move, this electric field polarization will produce a voltage which is proportional to the physical displacement.

Thus PVDF only generates and electrical pulse when there is a change in mechanical force on it. Since PVDF is manufactured in sheets it was easily attached to the model wings that were placed in the wind tunnel. By attaching an amplifier to the transductive film, it was possible to measure changes in lift.

Students conducting this experiment will learn about polyvinylidene fluoride and how to harness its piezoelectric properties. They will learn elementary electronics by assembling the amplifier needed with the PVDF.

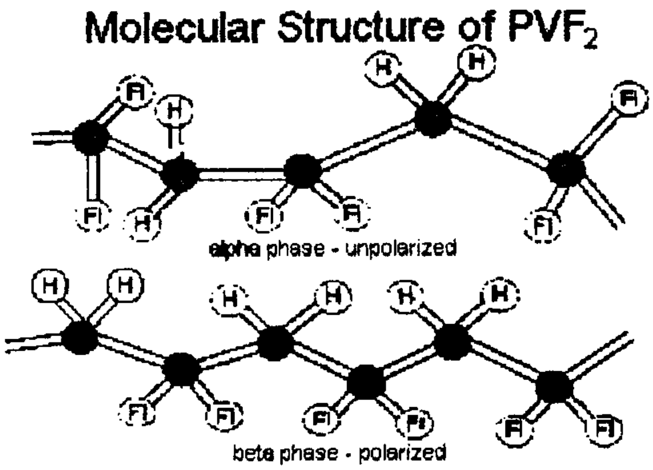

Figure 1

This experiment demonstrated an efficient and inexpensive method to measure wing stability by using polyvinylidene fluoride as the primary sensor device. A computer digitizer was also used to get the graphical results from the PVDF sensor. The procedures required to execute the experiment are listed below:

- design and construction of model wings with sensor mounted
- mounting of model wings in the wind tunnel
- design and construction of amplifier circuit and data collection

Design and construction of model wings with sensor mounted:

Four different wing designs were used (fig 2).

Procedure:

- The wings were constructed with a sturdy flexible plastic sheet. The sheet was shaped into the design for the wings and then forced in shape with cellophane tape and supported by holders at the ends. Balsa wooden mounts were attached to the ends of the wing for mounting (fig 3).
- The sensor was attached to the underside of the wing for lift measurement. The PVDF was attached with glue to the plastic. Electrodes were attached to the ends of the film.

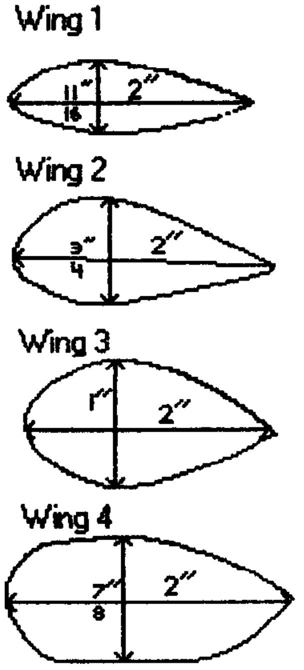

Figure 2

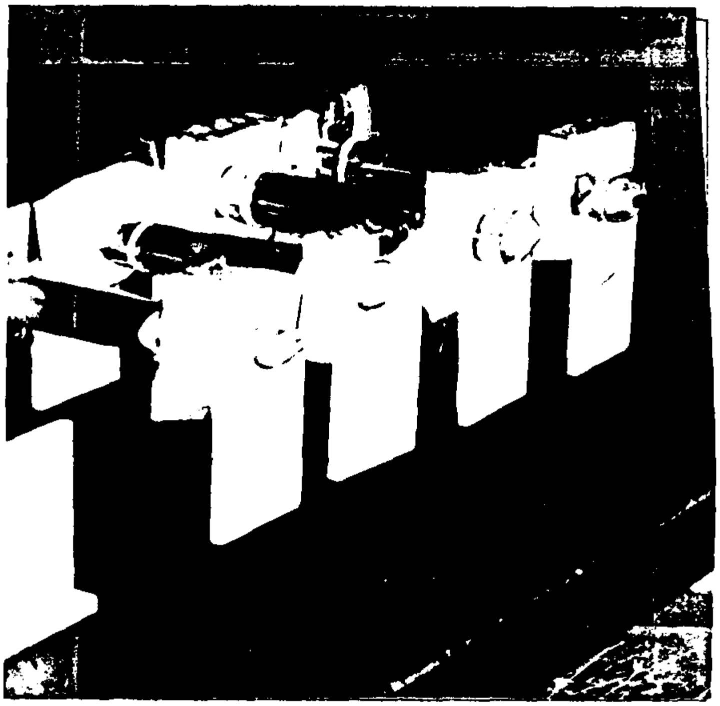

Figure 3

- To make contact with the PVDF sensor aluminum foil was placed between the film and the wing at one end and over the film on the other end. Rubber bands were placed over the overlap of the PVDF and aluminum to insure contact . The aluminum foil was then attached to wires which led to the amplifier.

Mounting of model wings in the wind tunnel

- A low speed, indraft wind tunnel was used to provide the laminar air flow around the wings. The wind tunnel is 22 feet long with a test area of 10in. * 10in. * 60in. The ratio of the contraction section to the test section is 1:20 with the diffuser section sloped at an angle of 7 degrees. Wind speeds range from 0 to 20.1 m/sec (45 mph) and is produced by two-variable speed fans.
- A mounting stand was constructed with walnut wood. The stand had rubber feet underneath it to increase friction between it and the wind tunnel. The stand was created so that wing models could be mounted and dismounted quickly. The stand was equipped with a compass so the angle of the wing could be measured.
- The stand with the model wing was placed in the wind tunnel with a weight placed on a lower part of the stand so that the wing remained stationary. Wires were connected with alligator clips to the aluminum foil. This made it easy to switch wing models in the wind tunnel (fig 4).

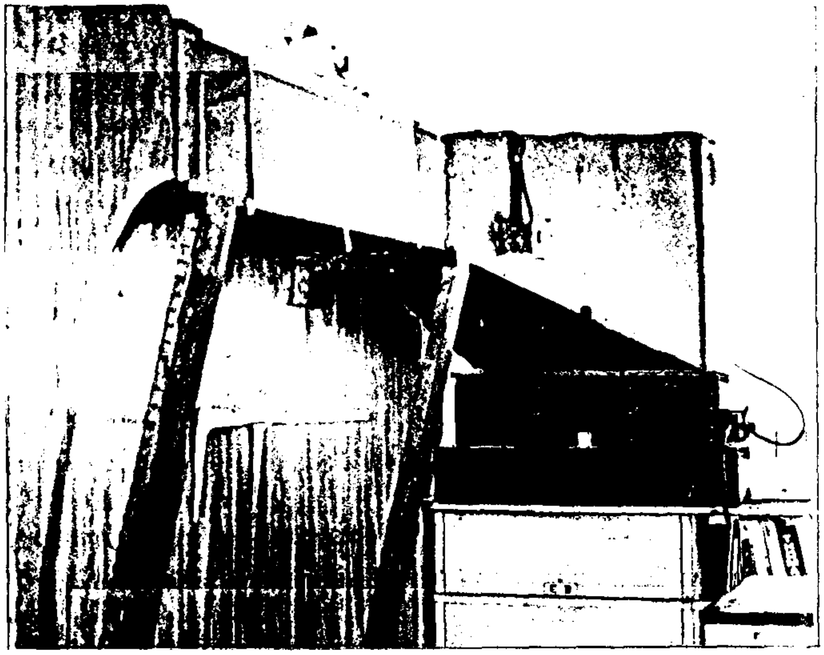

Figure 4

Design and construction of amplifier circuit and data collection:

- The amplifier circuit (fig 5) is used so that the pulses generated by the PVDF are able to be read by the CHAMP analog to digital converter interface.
- The CHAMP was connected to an Apple IIe computer.The data was then transferred to an IBM compatible computer and onto a spreadsheet which was used to generate the graphs.

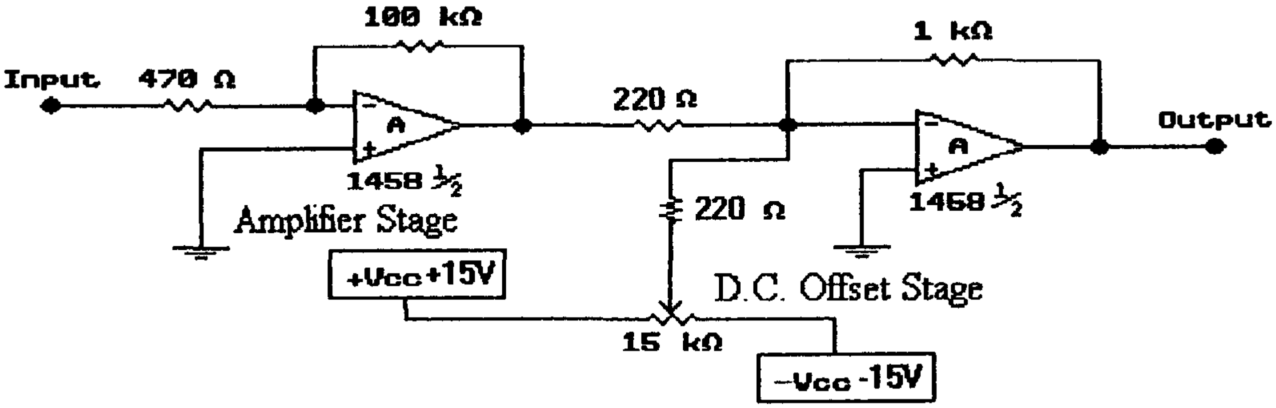

Figure 5

Results:

Bernoulli's principle occurs in wing mechanics because the distance air travels above a wing is more than the distance it travels underneath a wing. This means that the ratio of the length on the top a wing to the length underneath can be used to compare lift of wings. The lift of the four model wings are graphed below (fig 6). The higher the ratio the more lift the wing can produce.

The CHAMP analog to digital converter allows the electric pulses produced by the PVDF on the underside of the wing to be stored on a computer. By using a spreadsheet, graphs were created for each wing with approximately 45 MPH air flow (fig 8). Since PVDF only creates a pulse when a change in lift occurs, the graphs are alternating waves. To attain a qualitative value, the voltage ranges for each wing was compared (fig 7). The greater the range, the more unstable the wing is.

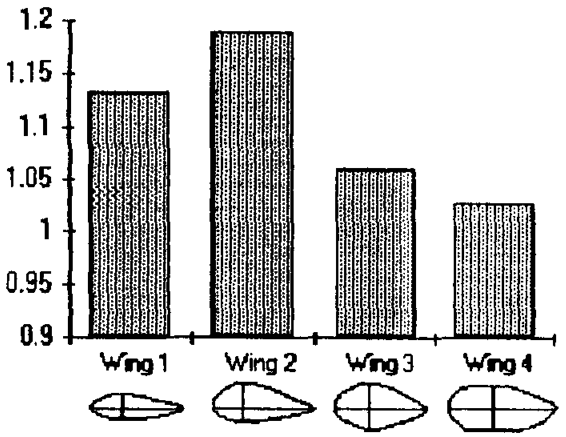

Figure 6: Relative Lift

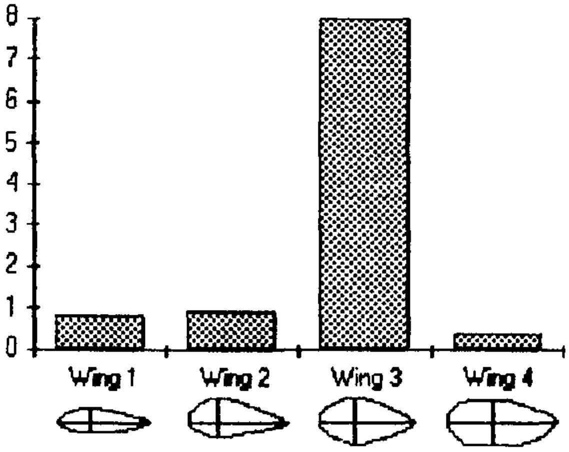

Figure 7: Relative Instability Range

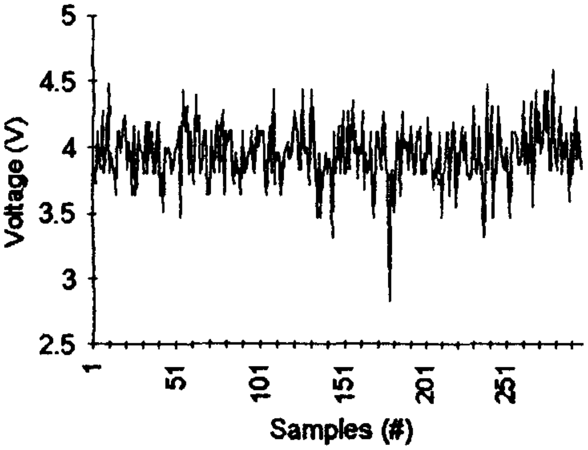

Figure 8a: Wing 1

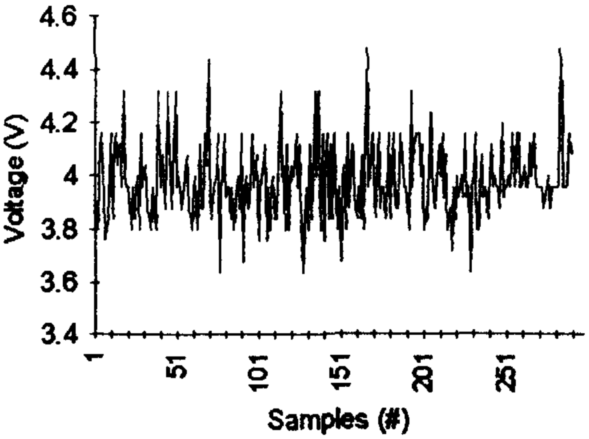

Figure 8b: Wing 2

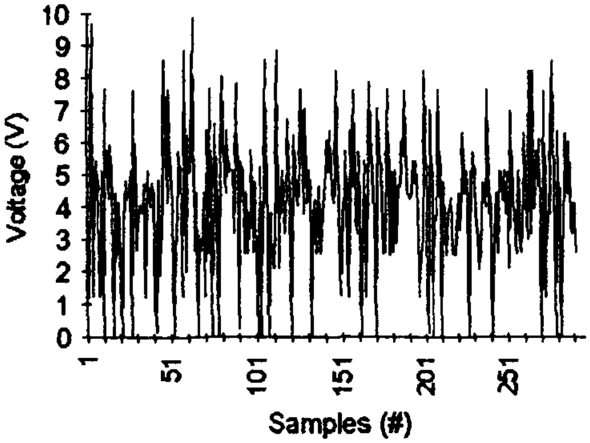

Figure 8c: Wing 3

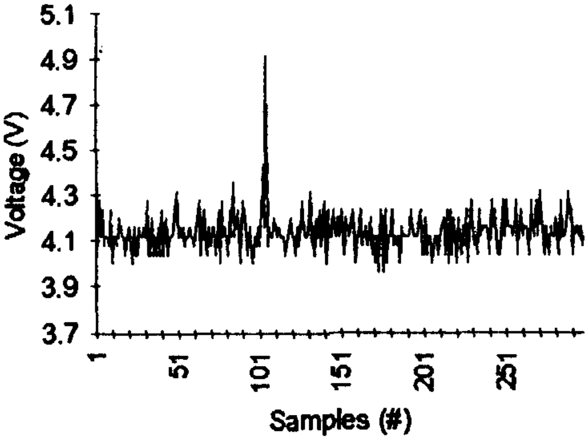

Figure 8d: Wing 4

Conclusions:

The stability tests indicated that wing 4 was the most stable of the models. However, the lift from wing 4 was lower than the other designs. This is because wing 4 had a "symmetrical" shape where the distance above the wing was very close to the distance below it. Its small thickness and its symmetrical shape was responsible for the high stability.

Stability for wing 1 was second best. The lift from this wing was also second best. The "streamline typical" shape of this wing help reduce drag and produce a substantial amount of lift with a large amount of stability.

Stability for wing 2 was close to that of wing 1. However, the lift of the wing was considerably more than that of wing 1. This is because the "typical" shape wing boasts a larger thickness which increases the distance above the wing. This will produce considerable stability and high amounts of lift.

Stability for wing 3 was eight times worse than the other models. This model was designed with the purpose to indicate a low stability rating. This "tear drop" shape produces a low amount of lift and is useful for comparison to the other models.

An Affordable Material Testing Device

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Participating Students: R. Clark, S. Stump, T. Flanders, J. Warner

Introduction:

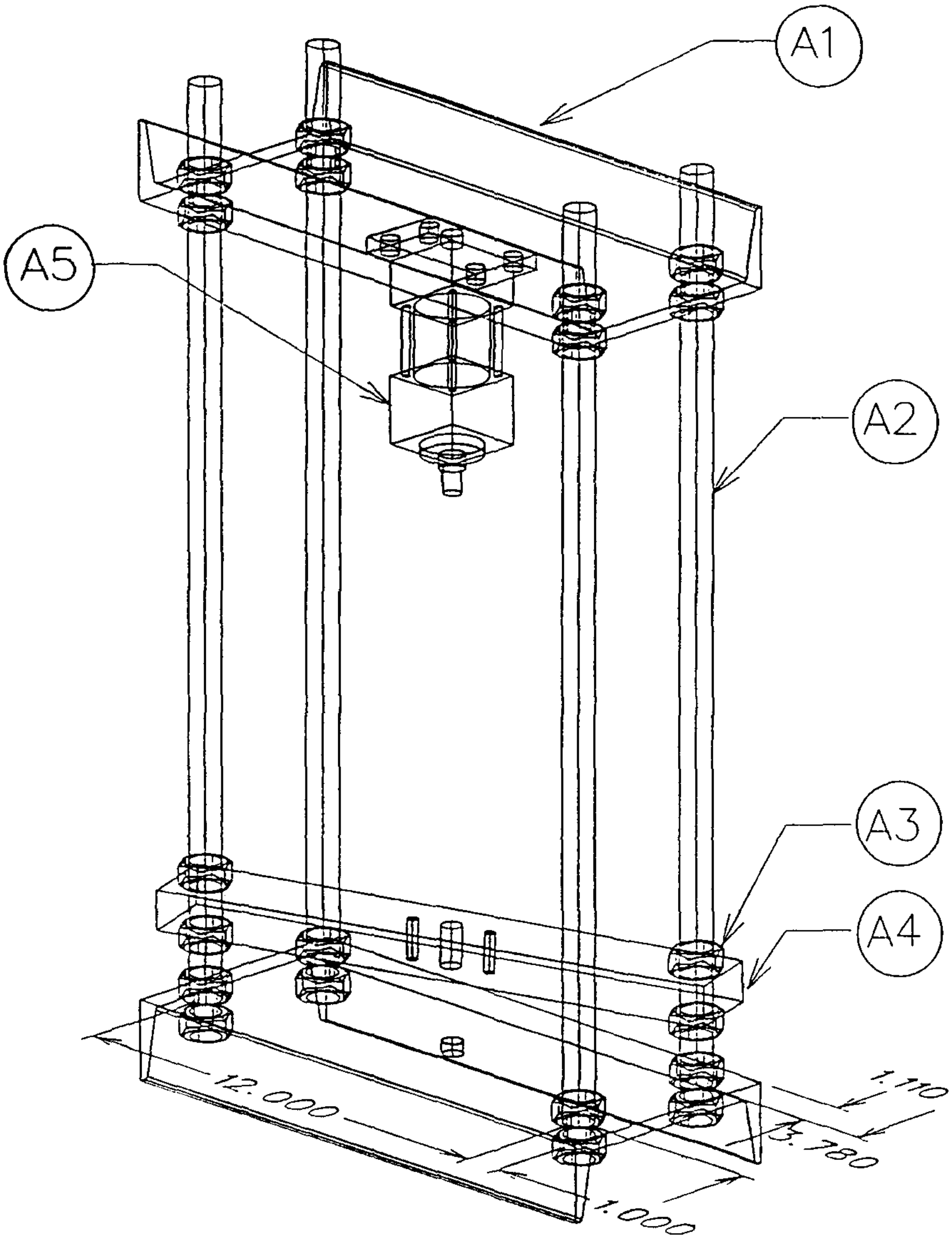
One of the thrusts in engineering technology education is to combine a laboratory experience with the theoretical portion of the coursework. Experimental demonstration is an important factor in creating interest and motivation in learning. Most of the time budgetary limitation does not permit the purchase of expensive laboratory equipment. The project presented here is an effort to design an universal testing device for the strength of materials laboratory at the minimum cost.

This project was assigned to a group of four students as a partial requirement for a sophomore level strength of materials course. The paper describes the construction of a testing apparatus for a number of experiments such as tensile test, punching shear test, column buckling, and spring rate.

The device consists of a metal frame made of two channel iron with one as the base and the other at the top supported by four all thread rods for height adjustment (fig 1). A hand operated hydraulic power pack is used to operate a cylinder that applies force on test samples. Applied force is determined by multiplying the pressure recorded with the effective area of the hydraulic cylinder. Accessories include a holding fixture for punching shear samples and two cup shaped holder for column buckling, and two jaws for holding samples for tensile tests.

After brief description of each experimental procedure and data collection the results are graphically reproduced at the end of this paper. This device is meant to be used for classroom demonstration and laboratory exercises. It is lightweight, simple and affordable.

This testing device will perform the following experiments:
- Punching Shear
- Spring Rate
- Tensile Test
- Beam Bending
- Column Buckling



ITEM	SIZE	MATERIAL	QTY	PRICE
A1	C 6 x 10.5 x 14"	Am.Std.Steel Channel	2	$0.69/Lb.
A2	3/4" - 10 x 24"	Steel Thd. Rod	4	$4.50 ea.
A3	3/4" - 10	Steel Zink Nuts	20	$0.40 ea.
A4	1" x 2" x 15"	Aluminum 6063	1	$51.14/8'
A5	62205K11 MF2	Dbl. Act. Hyd. Cyl.	1	$230.88
If everything was purchased:			Total Cost	**$898.71**

FIG - 1

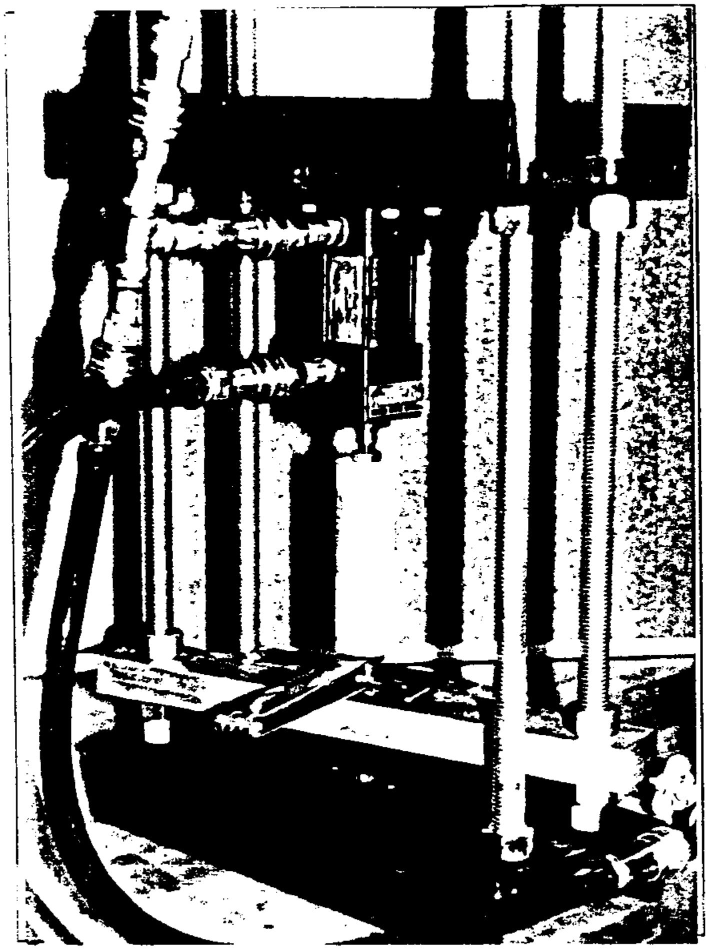


Figure 2

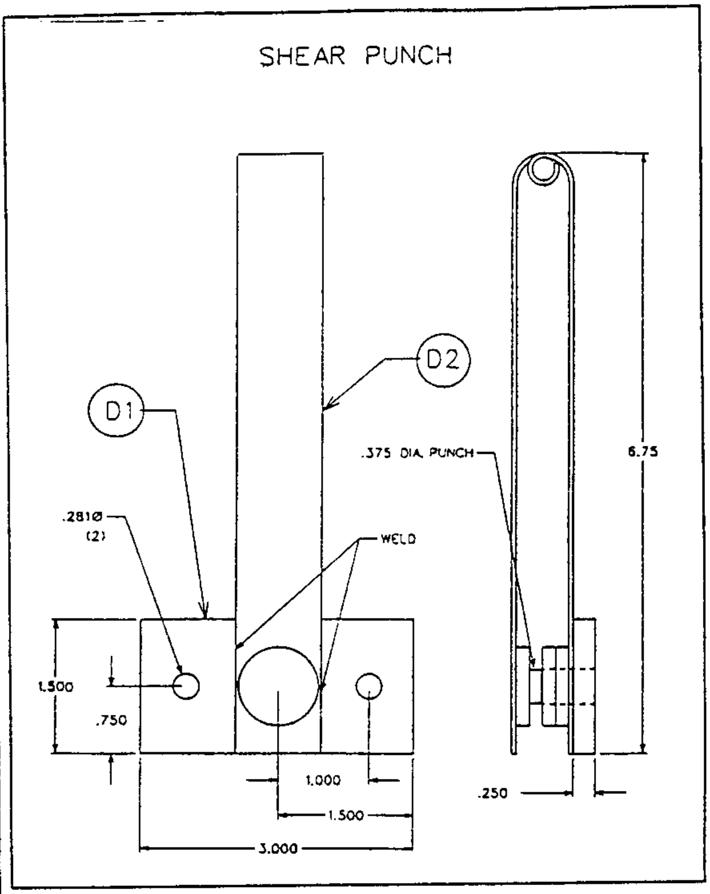


Figure 3

Punching Shear (fig 2 & 3):

The purpose of this experiment is to determine the ultimate shear strength of various specimens.

Procedure:

- Measure and record all specimen thicknesses
- Measure punch and die diameters
- Estimate force required to punch specimens
- Install punch and die and proper shaft on cylinder
- Insert specimen between punch and die
- Set directional valve to extend position
- Apply force with the hand pump till shear failure
- Lift punch out of specimen and die
- Determine maximum shear strength for all specimens

See figure 12 for the graph.

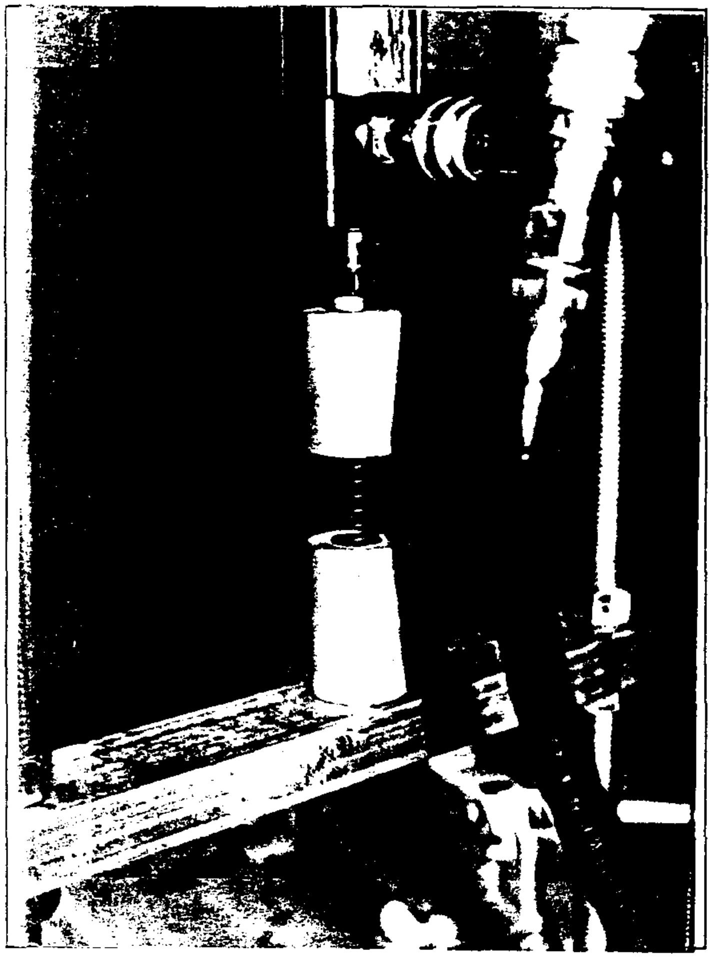
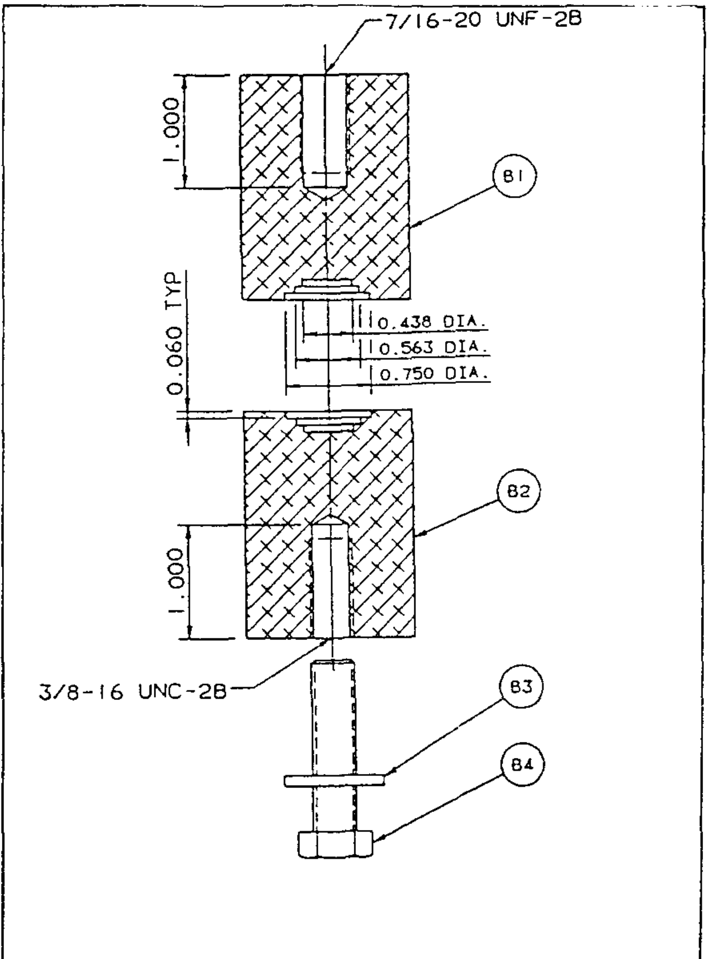


Figure 4



ITEM	SIZE	MATERIAL	QTY	PRICE
B1	1 1/2" dia. x 2" lg.	6061 Aluminum	1	$2.55
B2	1 1/2" dia. x 2" lg.	6061 Aluminum	1.	$2.55
B3	7/8" washer	Zinc plated steel	1/net 5lb	$8.11
B4	3/8" bolt	Zinc plated steel	1/net 100	$12.10
			TOTAL	$25.31

Figure 5

Spring Rate Test (fig 4 & 5):

The purpose of this experiment is to find rate of springs with various geometry.

Procedure:

- Find the inside and the outside diameter of each spring
- Measure the wire diameter of each spring
- Find free length of each spring
- Attach the upper half of the spring test fixture to the hydraulic cylinder and the lower half to the adjustable cross member.
- Place a spring into the fixture and slowly lower the hydraulic cylinder. Make sure that the spring sets inside of the recess of the fixture. Apply enough force to hold the spring in place.
- Set a dial indicator on the top or bottom of the upper spring fixture to measure the amount of deflection.

See figure 13 for the graph.

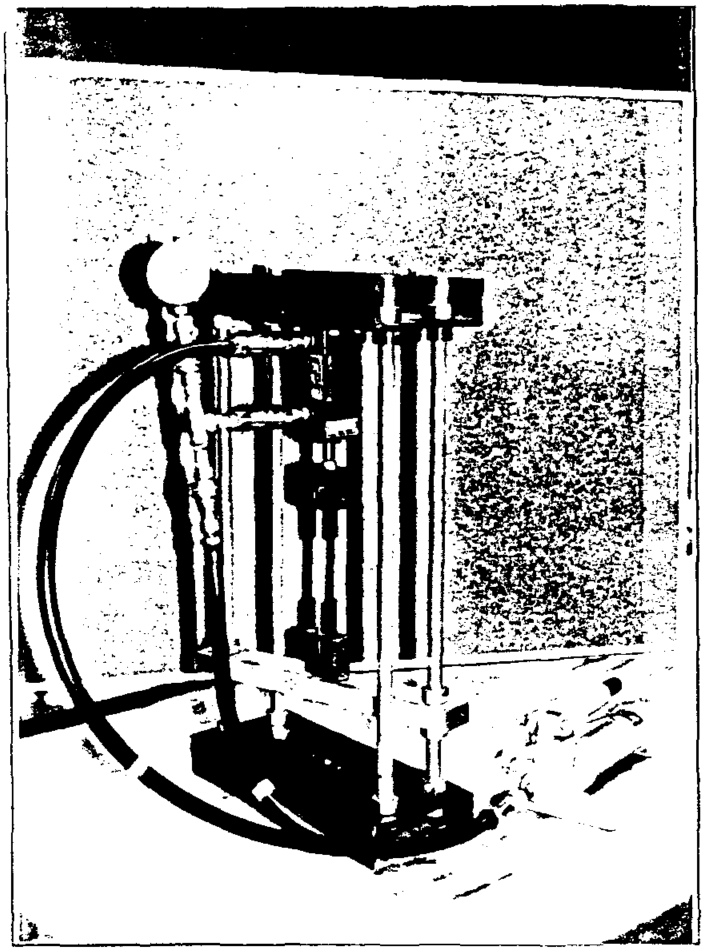
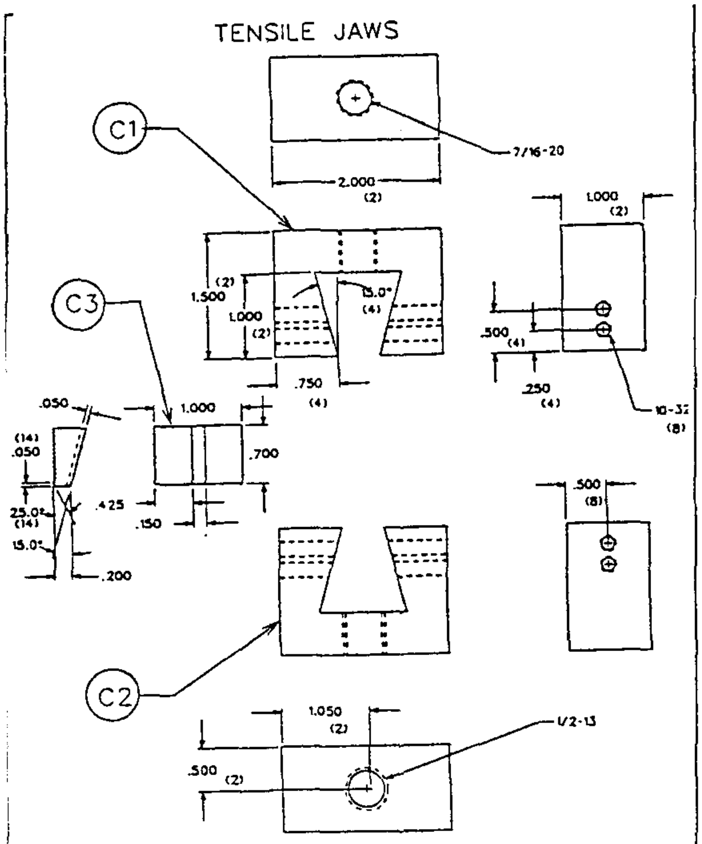


Figure 6



ITEM	SIZE	MATERIAL	QTY	PRICE
C1	1"x1.5"x2"	0-1 Steel RB-50 Upper Jaw Support	1	$42.91/36"
C2	1"x1.5"x2"	0-1 Steel RB-50 Lower Jaw Support	1	$42.91/36"
C3	.5"x.75"x1"	0-1 Steel Jaw RB-50	4	$42.91/36"
C4	10-32x.500 NS	Half Dog Set Screw	8	$14.81/100

Figure 7

Tensile Test (fig 6 & 7):

The purpose of this experiment is to obtain experimental data for a stress strain diagram.

Procedure:

- Attach the upper and lower jaws (fig 7) to the fixture and adjust the distance between the jaws if required.
- Using the dial calipers and micrometers, determine measurements of the sample.
- Place the sample in the tensile testing fixture. Make sure that it is properly aligned.
- Slowly increase the pressure with the hand pump, thereby increasing tension on the sample.
- Continue to increase the pressure and incrementally record your data until the sample ruptures.
- After the sample has ruptured remove it from the fixture, place the fractured ends together, measure and record the length.

See figure 14 for the graph.

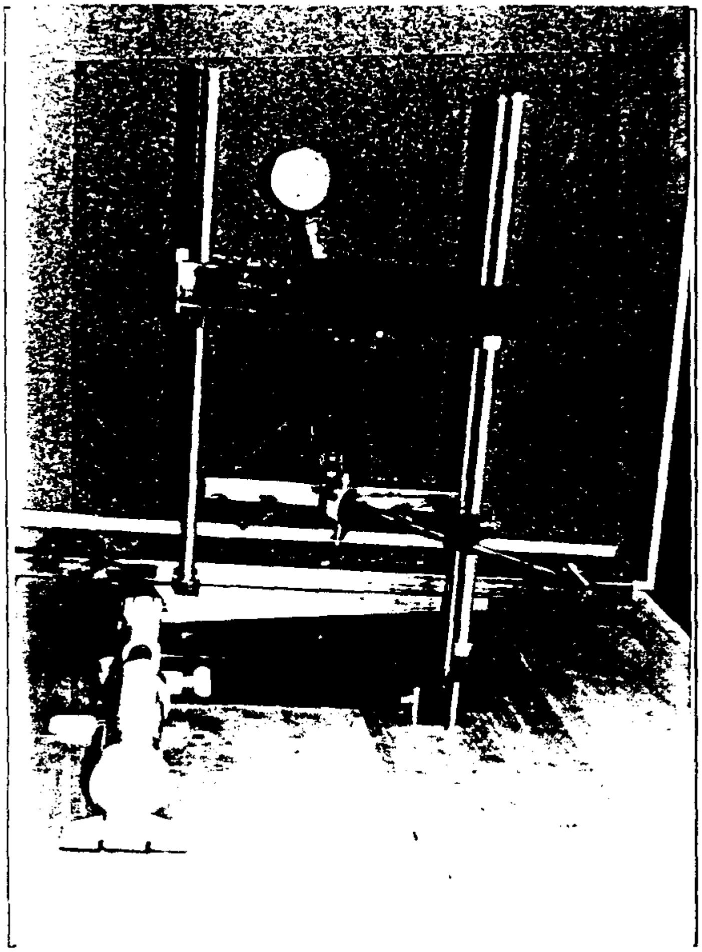
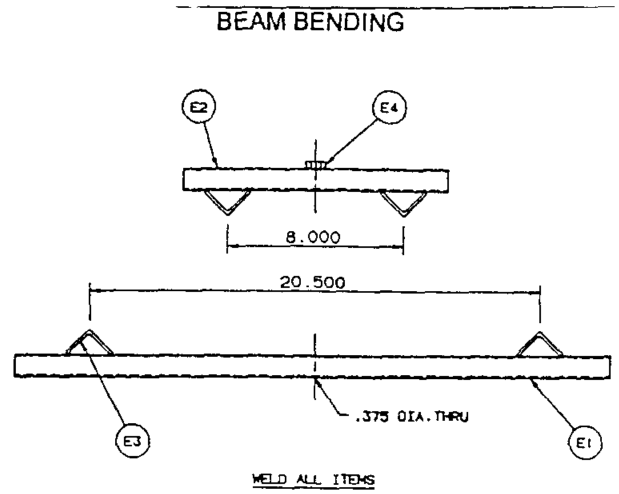


Figure 8



ITEM	SIZE	MATERIAL	QTY	PRICE
E1	1"x1"x16 ga.x27" lg.	Steel sq. tube	1	$2.87
E2	1"x1"x16 ga.x12" lg.	Steel sq. tube	1	$1.50
E3	1 1/2"x 1 1/2"x2 1/2" lg	Steel angle	4	$4.70
E4	7/16" nut	Zinc plated steel	1/net 100	$7.56
E5	1/8	Mild-steel	1/5 lb	$8.50
			TOTAL	$25.13

Figure 9

Beam Bending (Flexural Strain) Test (Fig 8 & 9):

The purpose of this experiment is to determine flexural strain in a beam. Flexural strain is directly related to the bending moment and beam geometry.

Procedure:

- Attach the beam bending fixtures to the base of the testing device and the hydraulic cylinder.
- Place the specimen on the fixtures (Fig. 9) so that it is centered and in contact with four points on the fixture.
- Set the dial indicator with modified arm extension in place to measure the beam deflection once a compressive force applied with the help of the hand pump.
- Read and record the pressure gage and the dial indicator.

See figure 15 for the graph.

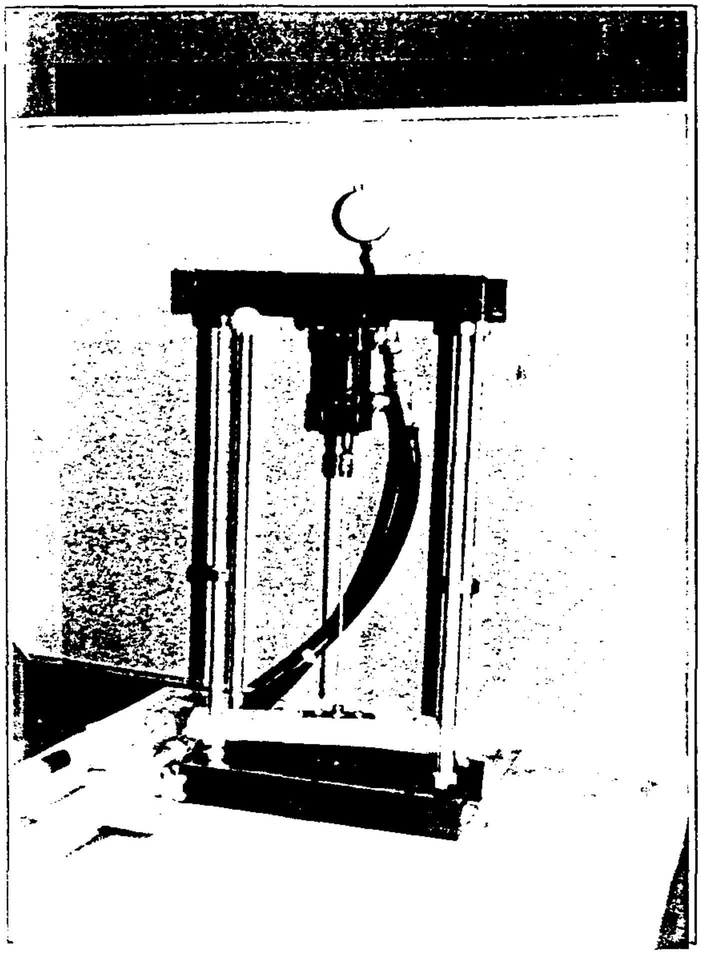


Figure 10

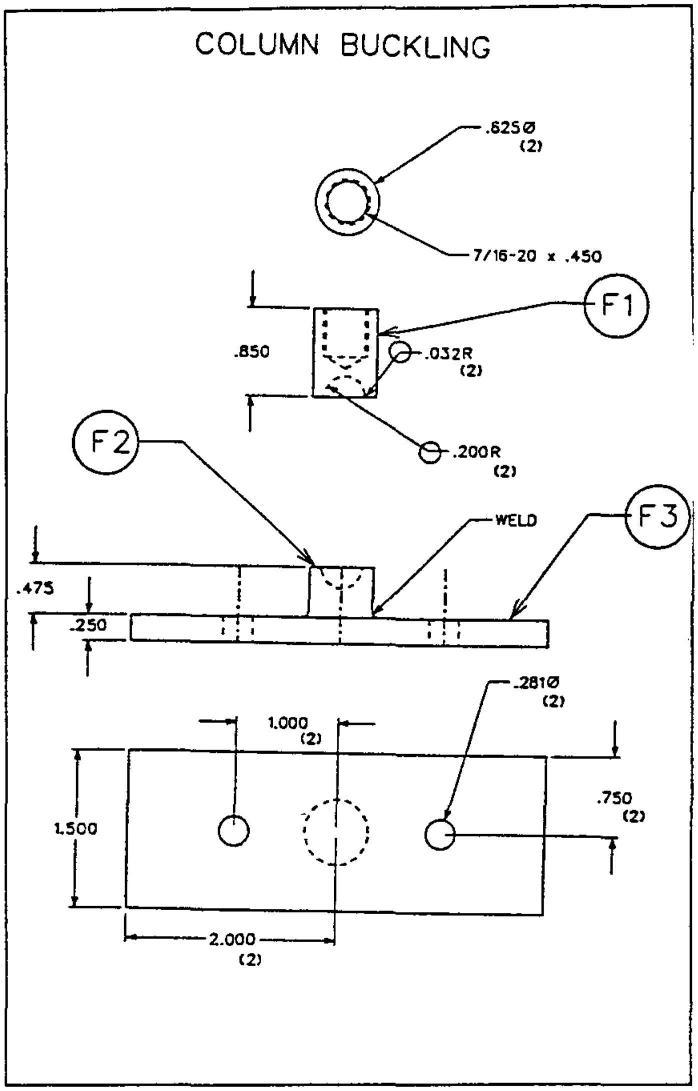


Figure 11

Column Buckling Test (fig. 10 & 11):

The purpose of this experiment is to determine the critical buckling load for slender columns.

Procedure:

- Measure and record the diameter and length of the specimen
- Oil both ends of the rod - insert into top seat.
- Extend cylinder rod using the hand pump.
- Continue to lower rod until seats hold the specimen rod.
- Slowly extend the cylinder rod - observe the specimen rod for bow and read the pressure.
- Calculate the critical load

Graphs:

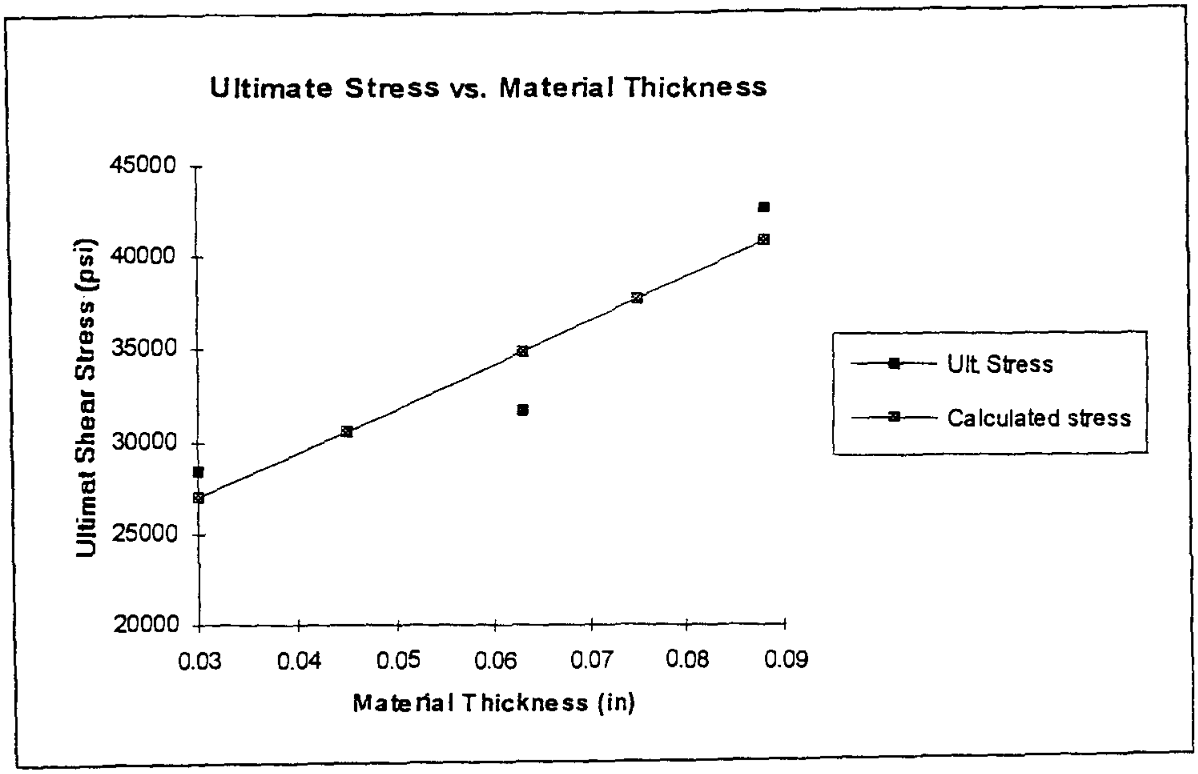


Figure 12 - Punch Shear: ultimate shear stress vs. material thickness

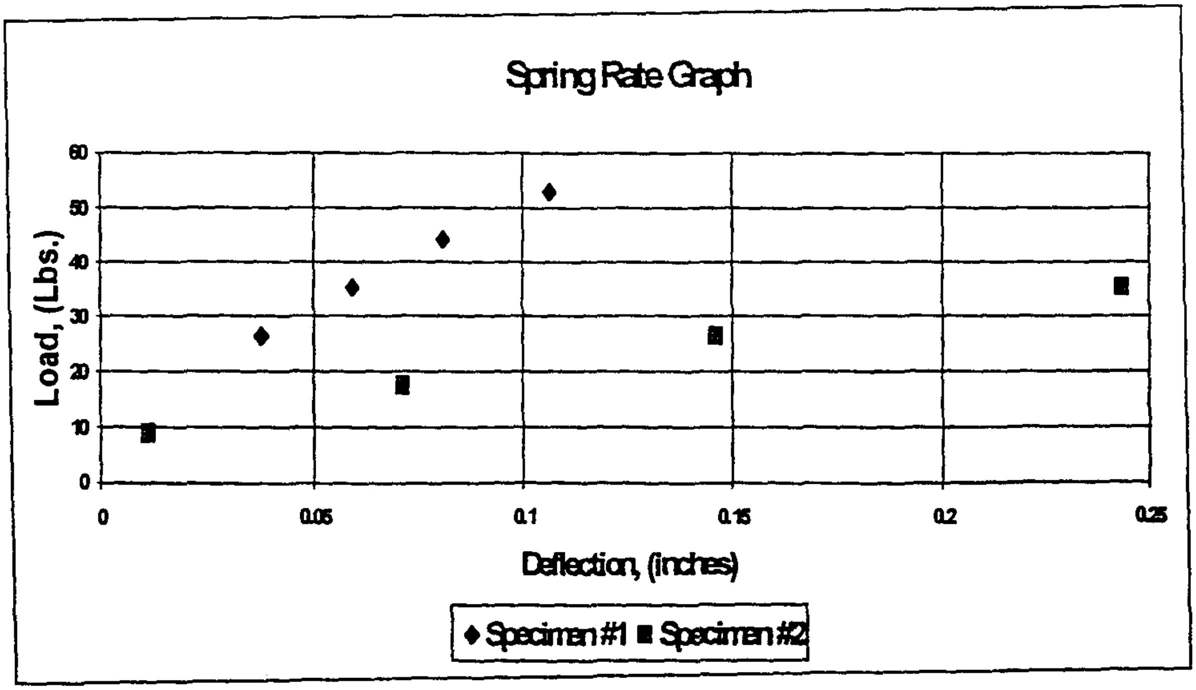


Figure 13 - Spring Rate: load vs. deflection of two sample springs

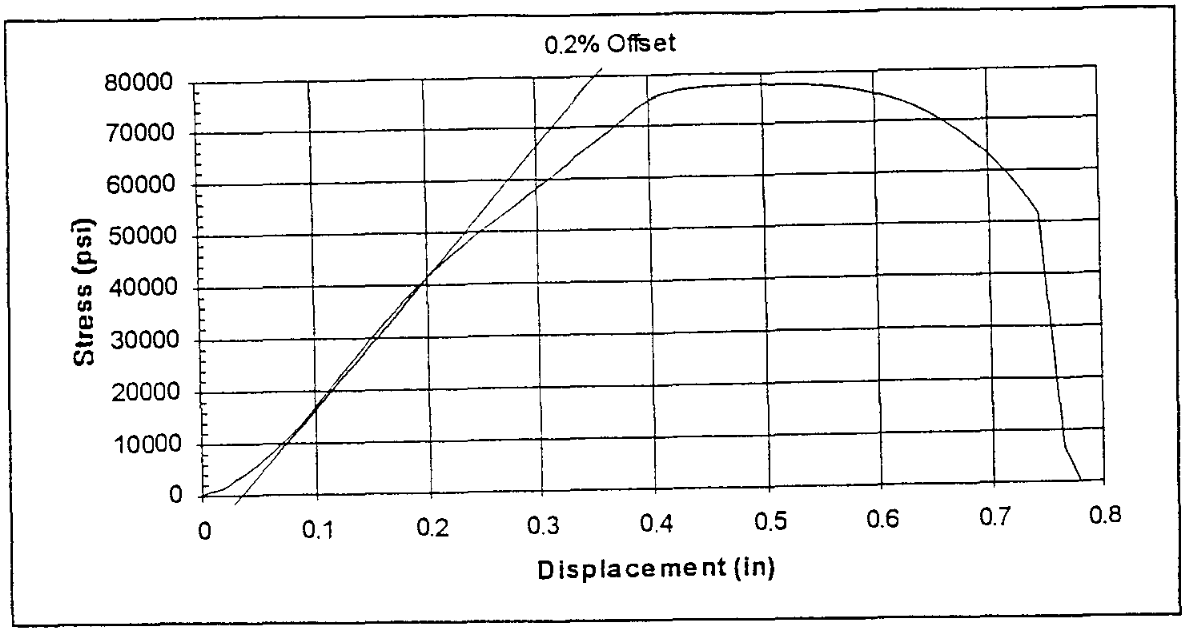


Figure 14 -Tensile: stress vs. displacement curve

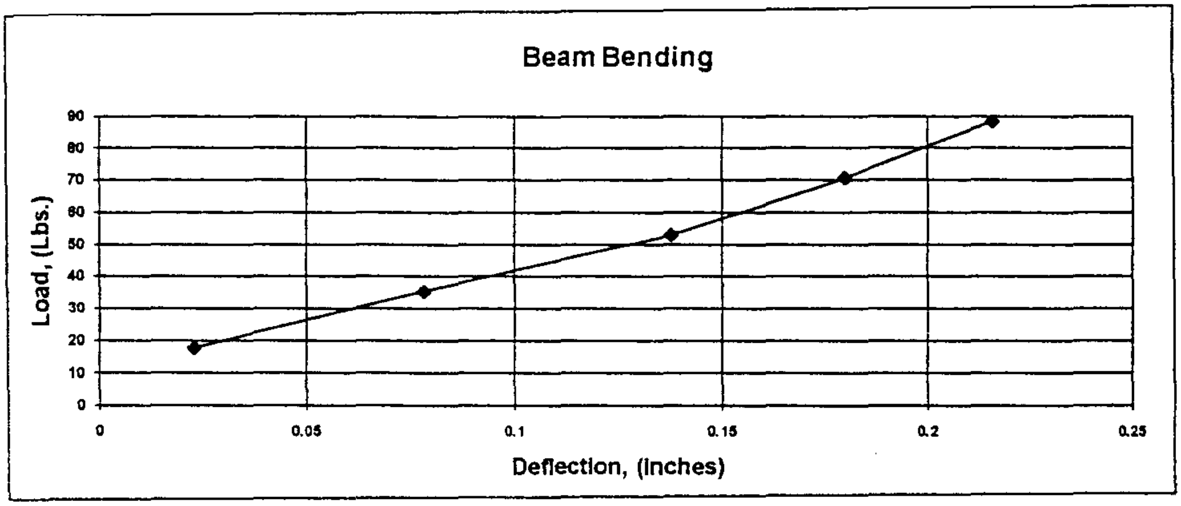


Figure 15 - Beam Bending: load vs. beam deflection

PHOTOCATALYTIC DESTRUCTION OF AN ORGANIC DYE USING TiO_2

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Photocatalytic Destruction of an Organic Dye Using TiO_2

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Key Words: Photocatalytic, Solar, Titanium Dioxide

Prerequisite Knowledge: Knowledge of basic semiconductor band theory and spectrophotometric techniques are helpful in understanding and performing the experiment.

Objective: To observe the effect of a photocatalyst activated by solar energy applied in the presence of a dye solution.

Equipment:

1. Basic glassware including 500 mL round-bottom flask, gas sparging tube, 1 cm x 20 cm drying columns, 500 mL gas washing bottle
2. Aquarium air pump
3. Magnetic stirrer
4. 10 mL plastic syringe
5. Bausch & Lomb Spectronic-20 UV-visible spectrophotometer (or similar instrument) and appropriate cells
6. Time measuring device
7. Soda Lime (P. K. Morgan Instrument Co.)
8. Desiccant (such as Drierite™)
9. 90%-saturated barium hydroxide solution
10. 0.45 µm nylon-membrane syringe filters (Gelman Sciences, Inc.)

Introduction:

Titanium dioxide, TiO_2, is used by college students on a regular basis whether they are aware of it or not. Widely known as the principle pigment in white paint, TiO_2 may be found as close as their eyelids and noses in the form of cosmetics and sunblock.

TiO_2 has recently attracted scientific attention due to photocatalytic properties in its colloidal and immobilized phases. Although the "chalking" of paint containing TiO_2 provided an early clue and sparked interest in its use as a photocatalyst(*1*), this property has found application recently in organic synthesis(*2*) and has been investigated as an environmentally clean method to destroy organics in water(*3,4*).

Colloidal anatase-TiO_2 is a semiconductor with a band-gap energy of 3.2eV which, when illuminated by light with wavelengths less than 360 nm will cause photoexcitation of the TiO_2 producing an electron and a positive hole, h^+. In the presence of dioxygen and water, hydroxy radicals ($OH\cdot$) are produced. In a solution containing an organic molecule such as a dye, the $OH\cdot$ will react with the molecule to produce carbon dioxide.

In this experiment, students use solar energy to activate an aqueous slurry of TiO_2 that mineralizes an organic dye to carbon dioxide. Since the solar energy spectrum possesses wavelengths below 360 nm, although at low intensity at the Earth's surface, the experiment described here can be performed in natural sunlight.

Procedure:

Apparatus:

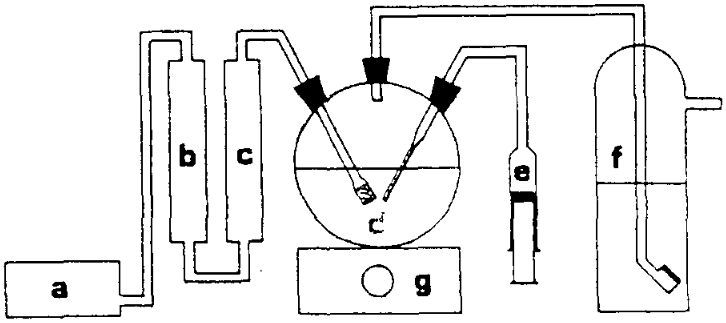

Figure 1. Schematic diagram of the reactor system. a. air-pump; b. drying column; c. soda lime column; d. 500 mL round-bottom flask; e. 10 mL sampling syringe; f. gas washing bottle; g. magnetic stirrer.

A schematic of the reactor system is shown in Figure 1. The reaction vessel is a 500 mL, 3-neck round bottom flask (Figure 1d). Ground-glass joints are secured with plastic clips available from Fisher Scientific Co. Air, which is pumped through the system using a Whisper-500™ aquarium air-pump (figure 1a), is dried through a 1 cm x 20 cm tube containing desiccant and the CO_2 removed using a similar tube containing "indicating" soda lime (P. K. Morgan Instrument Co.), Figure 1c. All connections are made with short lengths of latex tubing and secured with copper wire. The dry, CO_2-free air is introduced into the reaction vessel through a medium porosity, sintered-glass sparging tube attached through one of the necks of the flask. A 6 mm glass tube is inserted through a standard-taper 19/22 thermometer fitting for the collection of the effluent gases. The glass tubing is connected to a 500 mL gas-washing bottle containing 250 mL of 90%-saturated barium hydroxide (Figure 1f) fitted with a coarse sintered-glass bubbler.

Samples are collected via a 3.5 mm o.d. glass capillary extended into the solution through a #00 one-hole rubber stopper fitted into a standard-taper 19/22 thermometer fitting. Approximately 8 cm of 1/8" o.d. Tygon™ tubing is connected to the male end of a Luer-lock stopcock and a 10 mL syringe is attached to the stopcock. The entire apparatus is placed on a cork ring for support and the solution stirred with a magnetic stirrer.

Preparation of Solutions:

A 100 ppm stock solution of malachite green oxalate (MG) (J. T. Baker Chemical Co.) is prepared by dissolving 0.100 g of solid MG in a 1 L volumetric flask, diluting to the mark with water, and mixing well. The stock solution may be shared among many groups of students. Aliquots of 2, 5, 8, 10 mL are transferred to 100 mL volumetric flasks and diluted to give final concentrations of 2, 5, 8, 10 ppm for construction of a Beer's Law calibration plot. Simulated polluted water is prepared by diluting a 25 mL aliquot of stock MG solution to 250 mL in a volumetric flask to give a concentration of 10 ppm.

Air-free 90%-saturated solution of $Ba(OH)_2$ used for CO_2-analysis is prepared by stirring and heating a mixture of 30 g of solid barium hydroxide with 500 mL of water until the solid completely dissolves. The solution need not be heated above 35-40°C. The solution is allowed to cool to room temperature in a closed container to protect it from the air. When the solution is cool, ~450 mL of the clear solution is decanted into another bottle followed by 50 mL of boiled and cooled deionized water. The amount of barium hydroxide solution is sufficient for two complete runs of the experiment.

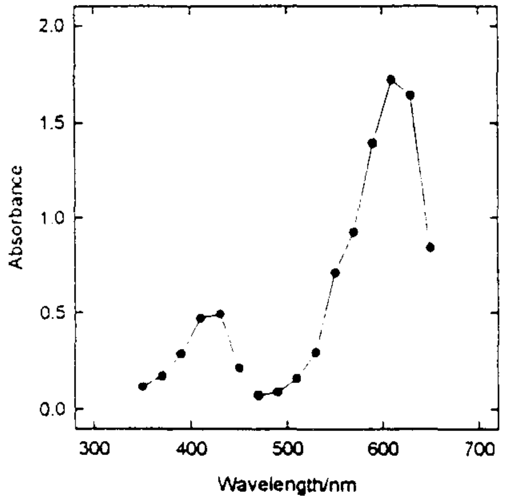

Figure 2. UV-visible absorbance spectrum of MG.

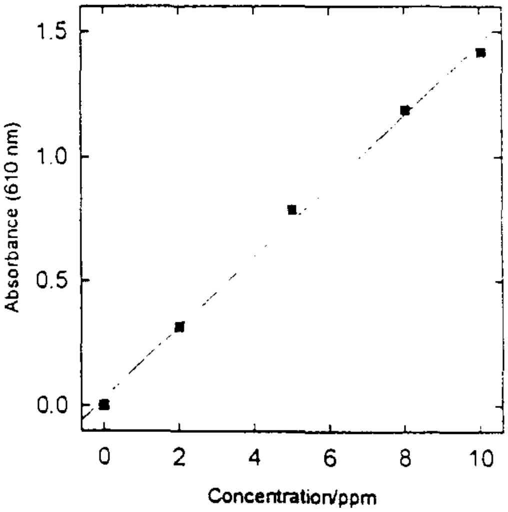

Figure 3. Beer's Law calibration plot of absorbance at 610 nm vs. concentration for MG.

Visible Light Spectrum and Beer's Law Calibration Plot

A Bausch & Lomb Spectronic-20 spectrophotometer may be used for the acquiring the UV-visible spectrum from 350 to 650 nm in 20 nm intervals on the 10 ppm MG solution using water as a reference (Figure 2). The Beer's Law calibration plot is prepared at 610 nm using the standard dilutions (Figure 3). If the wavelength of maximum absorbance differs from 610 nm, the wavelength measured on the particular instrument should be used.

Photocatalytic Reactor Set-Up and Acquisition of Samples

In order to isolate the effect of TiO_2, three different experimental treatments are used to study the decomposition reaction with MG; 1) a control experiment in which the solution is in contact with the catalyst but kept in the dark, 2) a second control experiment in which the solution is exposed to sunlight in the absence of the catalyst, and 3) conditions in which the solution is exposed to sunlight in the presence of the catalyst.

Set up the reaction system as shown in Figure 1. In experiments 1) and 3) a 0.1% suspension of TiO_2 (0.25 g TiO_2 per 250 mL solution) is used with a magnetic stirring bar to keep the catalyst suspended. The reaction vessel is completely covered with aluminum foil to prevent exposure to light prior to acquisition of the first sample. Place about 250 mL of the $Ba(OH)_2$ solution into the gas washing bottle and attach a short piece of latex tubing to the gas washing bottle from the reaction vessel as shown.

Experiments in the sunlight are performed during the early afternoon and initiated by removing the foil immediately after taking a 6 mL sample of the solution. Take additional samples every 15 minutes throughout the experiment, place in labeled test tubes, and protect from light with aluminum foil. Centrifuge samples for about 5 min and remove the remaining suspended TiO_2 by filtration using 0.45 µm pore-size nylon syringe-filter (Gelman Sciences, Inc.).

The effluent air containing CO_2 produced in the decomposition reaction passing through the gas washing bottle forms a precipitate of $BaCO_3$. The white solid $BaCO_3$ is collected by vacuum filtration on a fine, sintered glass filter, dried to constant weight in a 105°C oven for one hour, and weighed. The mass of CO_2 produced is determined from the mass of $BaCO_3$ formed.

Analysis of Data:

Compare the quantity of CO_2 produced to the theoretical mass of CO_2 which is calculated from the mass of MG placed in the reaction vessel. The concentration of MG for each sample is calculated from the equation of the Beer's Law calibration line. On one graph plot the concentration of MG vs. time for each of the experiments.

Effect of the Photocatalyst TiO_2:

Based on the results in the three experiments comment and explain your reasoning on the effect of solar energy and the photocatalyst TiO_2 in the reaction. Calculate the % yield of CO_2.

Notes to Instructor:

1. The % CO_2 found will be slightly low since some solution is removed for sampling during the experiment, but the error is less than 10%.
2. We suggest the zero-time samples be taken from the reaction vessel after the addition of the catalyst and just prior to exposing the vessel to the sunlight.
3. The initial concentration measured spectroscopically was consistently lower than the calculated concentration based on mass because of small amounts of stray light and adsorption of the dye to the surface of the catalyst.
4. A team of at least three students is recommended for the experiment which require should be performed over two laboratory periods. The first to prepare solutions, set-up and test the reactor system, and perform one of the control experiments.
5. This basic experiment can be extended by using other dyes and/or performing a kinetic analysis depending on the level of the students chemistry background.

References:

1. Kennedy, D. R.: *Trans. Far. Soc.* vol. 54, 1958, pp. 119.
2. Fox, M. A.: *Acc. Chem. Res.* 1983, vol. 16, pp. 1242.
3. Ollis, D. G.: *Environ. Sci. Technol.* vol. 19, 1985, pp. 480.
4. Schiavello, M. Ed.: *NATO Adv. Study Inst. Ser.* 1988, pp. 237.

ULTRASONIC WELDING OF RECYCLED HIGH DENSITY POLYETHYLENE (HDPE)

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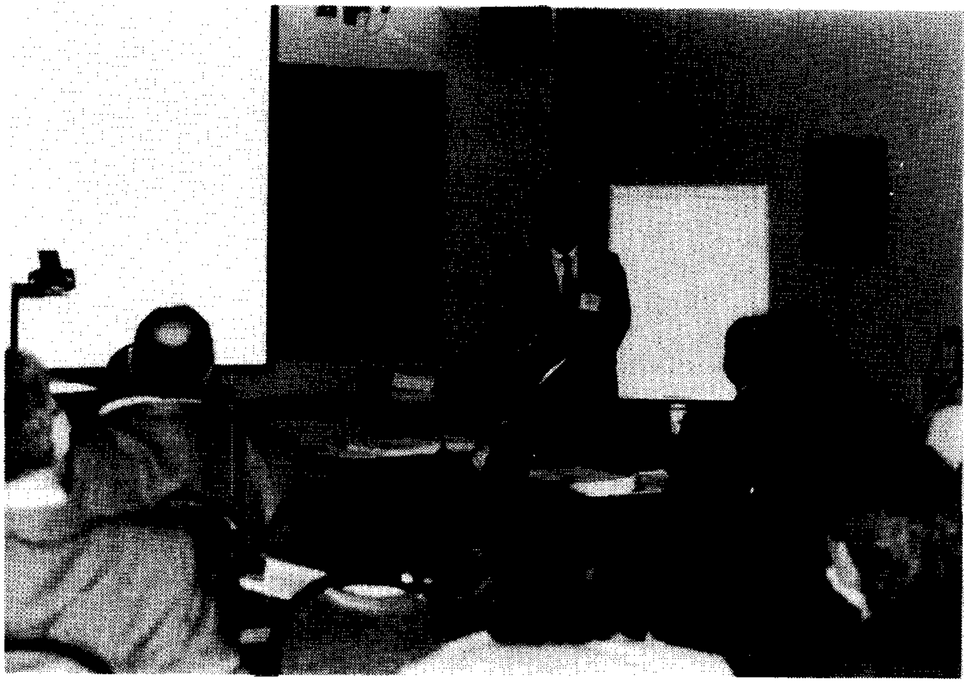

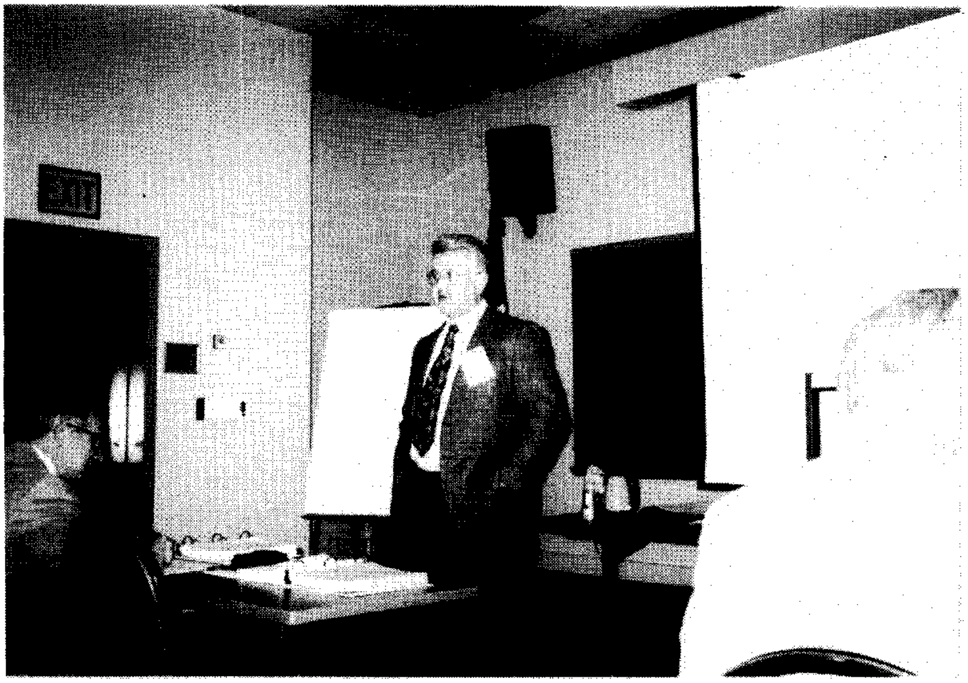
EXIT

Ultrasonic Welding of Recycled High Density Polyethylene (HDPE)

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KEY WORDS: ultrasonic welding, high density polyethylene, milk container, ASTM standards, tensile test, welding strength.

PREREQUISITE KNOWLEDGE: The students should understand the fundamentals of ultrasonic welding of thermoplastics and mechanical property testing of materials. The ability to use ASTM standards is also necessary for designing material test specimens and testing procedures.

OBJECTIVES: To understand the concept of thermoplastic ultrasonic welding and applications in industry.

EQUIPMENT AND SUPPLIES:

1. Recycled plastic one-gallon milk containers
2. Ultrasonic welding system
3. Steel rules and compass (or a specimen template)
4. Scissors
5. Tensile test machine.

INTRODUCTION:

Ultrasonic assembly is a fast, clean and efficient method of assembling or processing rigid thermoplastic parts, films or synthetic fabrics. Various ultrasonic assembly techniques are used by all segments of industry to join plastic to plastic, plastic to metal parts or other non-plastic material. Ultrasonic welding can replace or preclude the use of solvent, adhesives, mechanical fasteners, or other consumable. Ultrasonic welding is one of the techniques commonly used in industry.

The basic principle of ultrasonic welding is presented as follows. When two pieces of thermoplastics are vibrated together fast enough with appropriate amplitude, heat will be developed. As a result, a flow of plastic will occur at the joint interface. The method can provide strong and hermetically sealed components at a high production rate.

Figure 1 illustrates the principle of ultrasonic welder. A solid-state power supply converts 50/60 Hz current to 20 kHz or 40 kHz electrical energy. This high frequency electrical energy is supplied to the converter that changes electrical energy into mechanical vibratory energy at ultrasonic frequencies. The vibratory energy is then transmitted through an amplitude-modifying device called booster to the horn. The horn is an acoustic tool that transfers this vibratory energy directly to the parts being welded.

The variables in the ultrasonic welding include pressure, trigger force, weld time, hold time, after-burst (AB) delay and AB time. The characteristics of thermoplastics, such as melt temperature, modulus of elasticity and structure, determine the energy requirements of the weld. Optimum parameters can be identified through experiments according to the energy requirements.

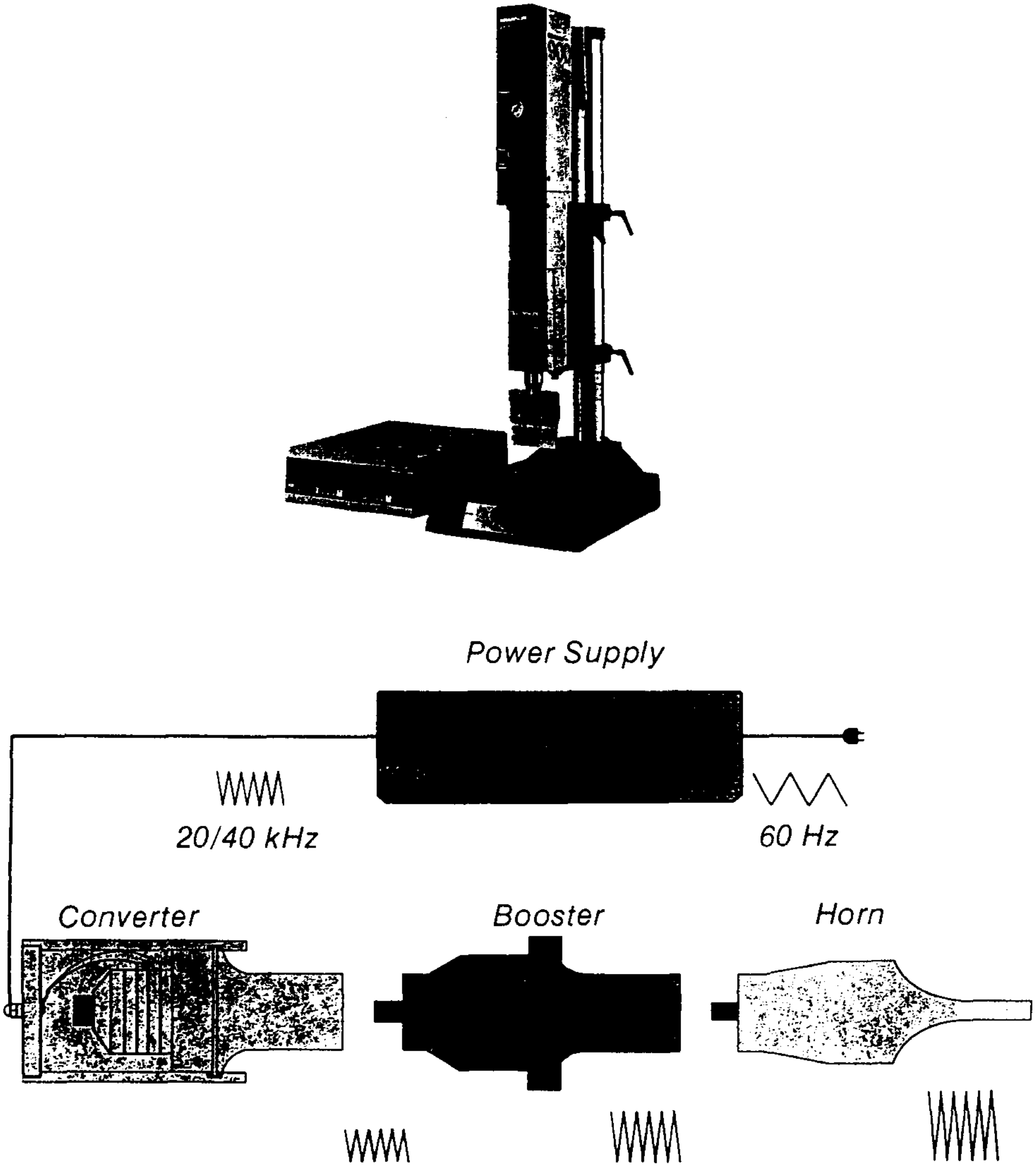

Figure 1 Principle of ultrasonic welding equipment (Courtesy Branson Ultrasonics Corp.).

PROCEDURE:

1. Collect, clean and dry recycled plastic milk containers.
2. Cut flat sheets of high density polyethylene from the milk containers. The sheet size is approximately 127 × 127 mm (5 × 5 in.).
3. Set parameters of the ultrasonic welding system as follows:

   Air pressure: 172 kPa (25 psi)
   Trigger force: 178 N (40 lb., dial reading 5)
   Down speed: Dial reading 5
   Hold time: 0.42 s
   Weld time: Variable, 0.21-0.33 s
   AB delay: 0.25 s
   AB time 0.05 s

4. Weld the plastic sheets to obtain proper quality.
5. Cut the tensile test specimens from the welded plastics according to dimensions specified by ASTM standards [1], as shown in Figure 2.
6. Test the tensile strength of the welding and evaluate the effectiveness of welding

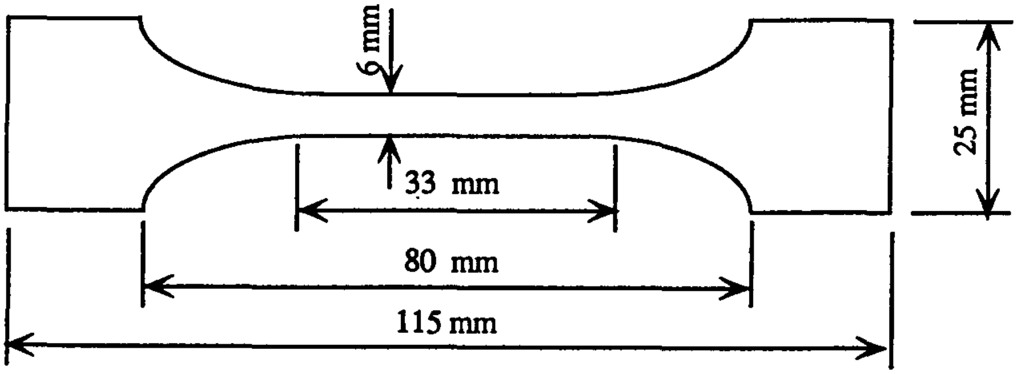

Figure 2 Dimensions of the tensile test specimen per ASTM standard D 638M-89.

SAMPLE DATA SHEETS

The data in the following table are tensile test results of welding joints formed with different weld time. All other parameters were identical.

Welding Time (s)	Peak Load N (lb.)
0.21	52 N (11.7 lb.)
0.24	817 N (183.8 lb.)
0.27	544 N (122.4 lb.)
0.30	426 N (95.9 lb.)
0.33	279 N (62.7 lb.)

INSTRUCTOR NOTES:

Students can perform investigative studies of the effect of welding parameters such as weld time on the weld quality. Figure 3 shows variation of peak load with ultrasonic welding time for recycled high density polyethylene welds. All other processing parameters were kept the same as those showed in step 3 of procedure section. Initially, the welding strength increased with the welding time up to a maximum at a weld time of 0.24-0.25 seconds. Then, the welding strength declined with increased weld time. It is concluded that the weld time of 0.24-0.25 second will provide the optimum welding strength for the thickness of high density polyethylene.

Due to the irregular weld section, it is a better choice to evaluate the peak load than shear stress for practical welding application.

REFERENCES:

1. ASTM: 1993 Annual Book of ASTM Standards, Vol. 8.01 Plastics (I), American Society for Testing and Materials, Philadelphia, 1993.

2. M. L. Berins: SPI Plastics Engineering Handbook (5th ed.), Van Nostrand Reinhold, New York, 1991.

3. Branson Ultrasonics Corp.: Ultrasonic Plastic Assembly, 1994.

SOURCES OF SUPPLIES:

The high density polyethylene was obtained from milk containers recycled by faculty and students without any cost.

ACKNOWLEDGMENT:

The donation of the ultrasonic plastic welder from Branson Ultrasonic Corporation, Danbury, CT is greatly appreciated.

Part of the financial support for this project is from the Illinois Department of Energy and Natural Resources through the Office of Solid Waste Research at the University of Illinois at Urbana-Champaign. Another part of the material is based upon work supported by the National Science Foundation under Grant No. DUE-9350629.

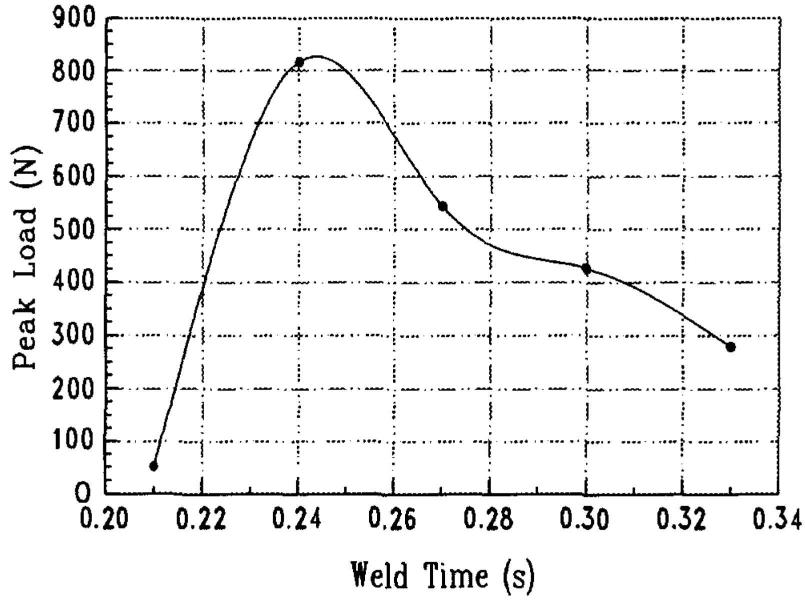

Figure 3 Variation of peak load with ultrasonic welding time for recycled high density polyethylene.

INTRODUCTION TO CONTINUOUS FIBER CERAMIC COMPOSITES

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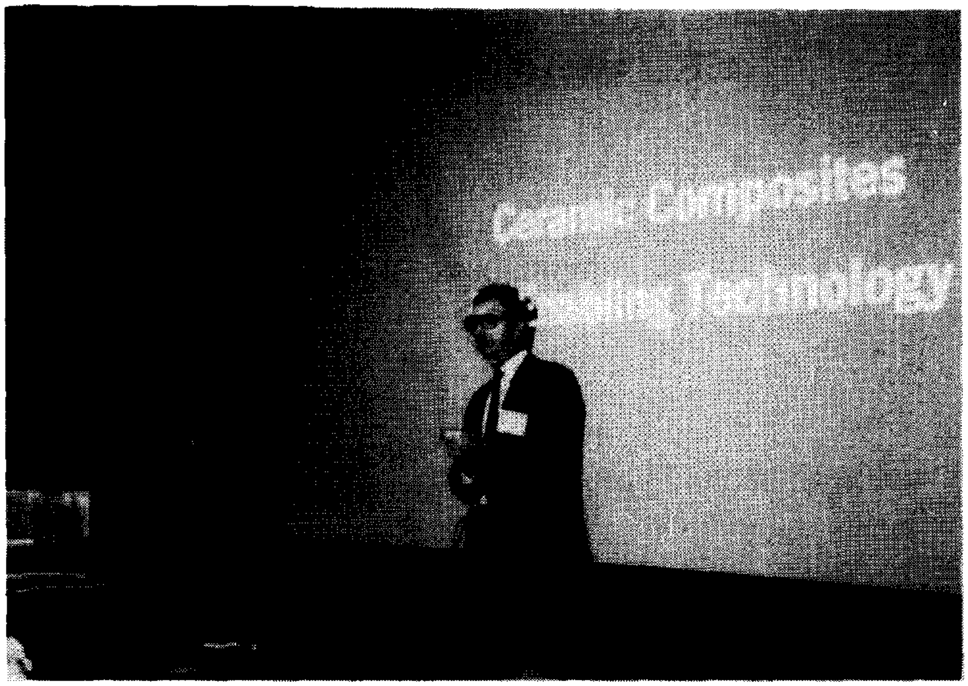
Ceramic Composites
Enabling Technology

INTRODUCTION TO

CONTINUOUS FIBER CERAMIC COMPOSITES

WILLIAM G. LONG
BABCOCK & WILCOX

Examples of Composites in Nature

- **Wood**
  - Cellulose fiber in a resin matrix
  - Probably the oldest, cheapest, and most reliable composite material
  - Limited in fracture stress level, temperature and geometry
- **Bone**
  - Collagen fibers provide toughness and flexibility

Ceramics Have Excellent High–Temperature Properties

- Strength to 1300 – 1400°C (2372 – 2552°F)
- Corrosion resistance
- Select from a variety of materials that include:
  - Carbides
  - Nitrides
  - Oxides

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Monolithic Ceramics Have Limitations

- Fail in brittle fracture
- Reliability suspect
- Thermal and mechanical shock properties poor

Definition of Terms

• Fiber	Normally used in a multi-filament tow, with 300 – 1000 filaments per tow. Each filament typically is about 10 microns diameter. Fibers may be carbide, oxide, or nitride.
• Matrix	Body of the composite that contains the fiber reinforcement
• Interface	Area in a composite where the fibers and matrix meet. The chemistry of the interface is critical to performance at temperature, and will control the pullout of the fiber at fracture.
• Fiber coating	Filaments are coated to produce an interface that allows pullout and protects the fiber from chemical attack

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CFCCs — An Engineered Solution

Fiber reinforcement retards crack propagation and provides mechanical support at high stress levels. The individual fibers must fail before fracture can occur.

Application Specific Properties

Like other composites, CFCCs offer great potential to customize materials for specific applications based on appropriate choices of constituents and design of fiber architectures

- Directional properties
- Controlled failure modes
- Tailored environmental stability

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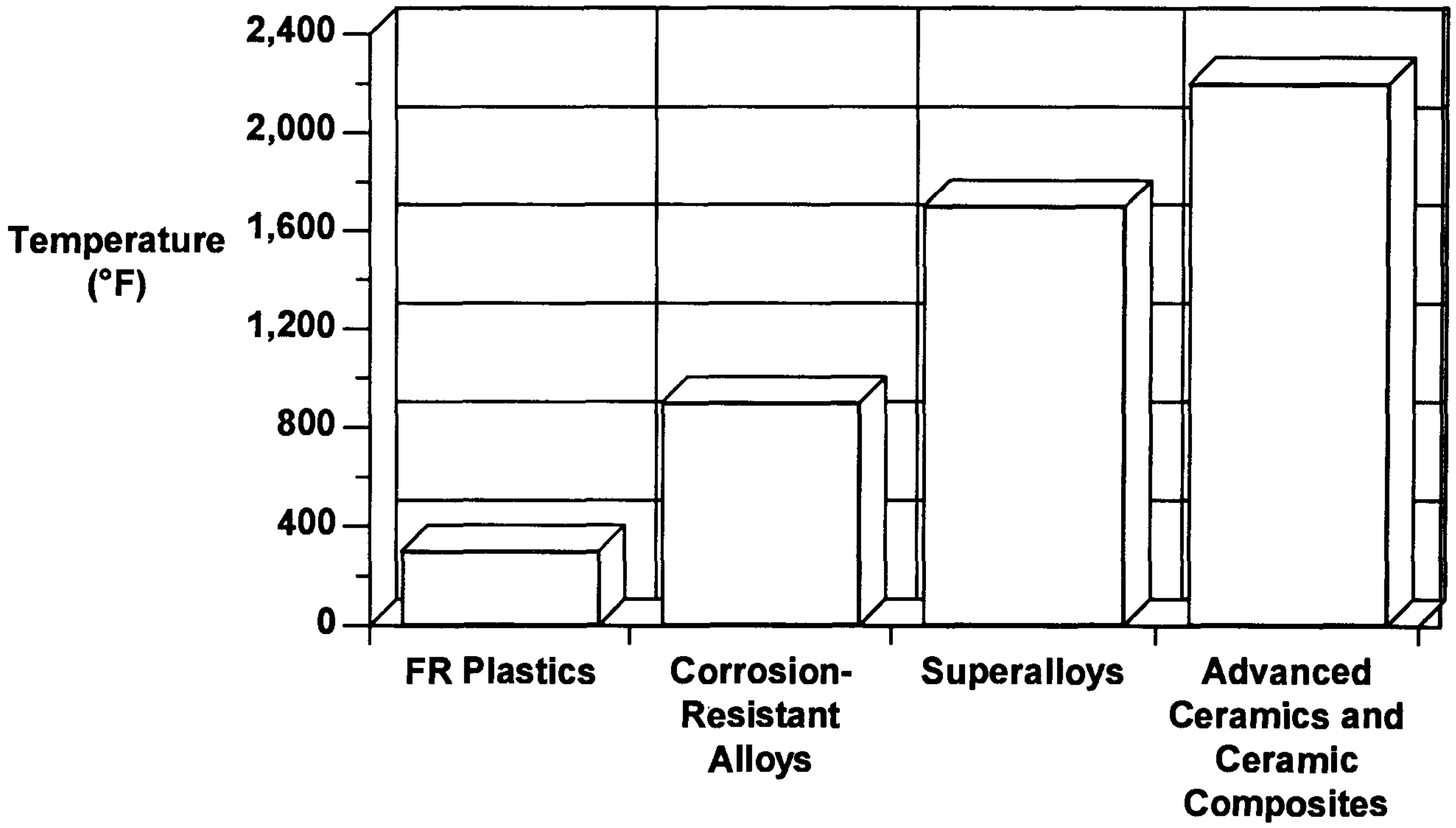
Potential Operating Temperature Regimes Based on Corrosion in Waste Incinerators



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Heat Exchangers

- **Performance targets**
  - Component temperatures to 1200°C (2192°F)
  - Proportional limit (matrix microcracking) >41 MPa (>6 kpsi)
  - Thermal conductivity >8.6 w/mK
  - Thermal shock resistance >330K in 5 minutes
  - Permeability negligible (to be determined)
  - Lifetimes of 24,000 hours to 10 years
- **Economic target**
  - ~$50 per foot

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Gas Turbine Components

- **Performance targets**
  - Component temperatures to ~1300°C (2372°F)
  - No film air cooling
  - Lifetimes of 24,000 hours
- **Economic target**
  - Ceramic hardware should be $\leq$ 3 – 5 times current metal hardware

DOE CFCC Program Components

- Porous radiant burners
  - Surface mat
  - Reverberatory screen
- Atmospheric–pressure and high–pressure heat exchangers
  - Tubes
  - Elbows
  - Manifolds
- Hot furnace fans
- Hot gas filters (for coal combustion and chemical processes)
  - Candle filters
  - Tubular filters

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DOE CFCC Program Components (Contd.)

- Gas turbines (large and small)
    - Combustor liners
    - Turbine tip shrouds
    - Turbine blades
    - Vanes
    - Blisks (bladed discs)
- Hot gas transfer pipes
- Radiant burner tubes
- Immersion heaters
- Boiler components
- Diesel engine valve guides and piston rings
- Steam reformer tubes
- Containment shell for sealless chemical pump

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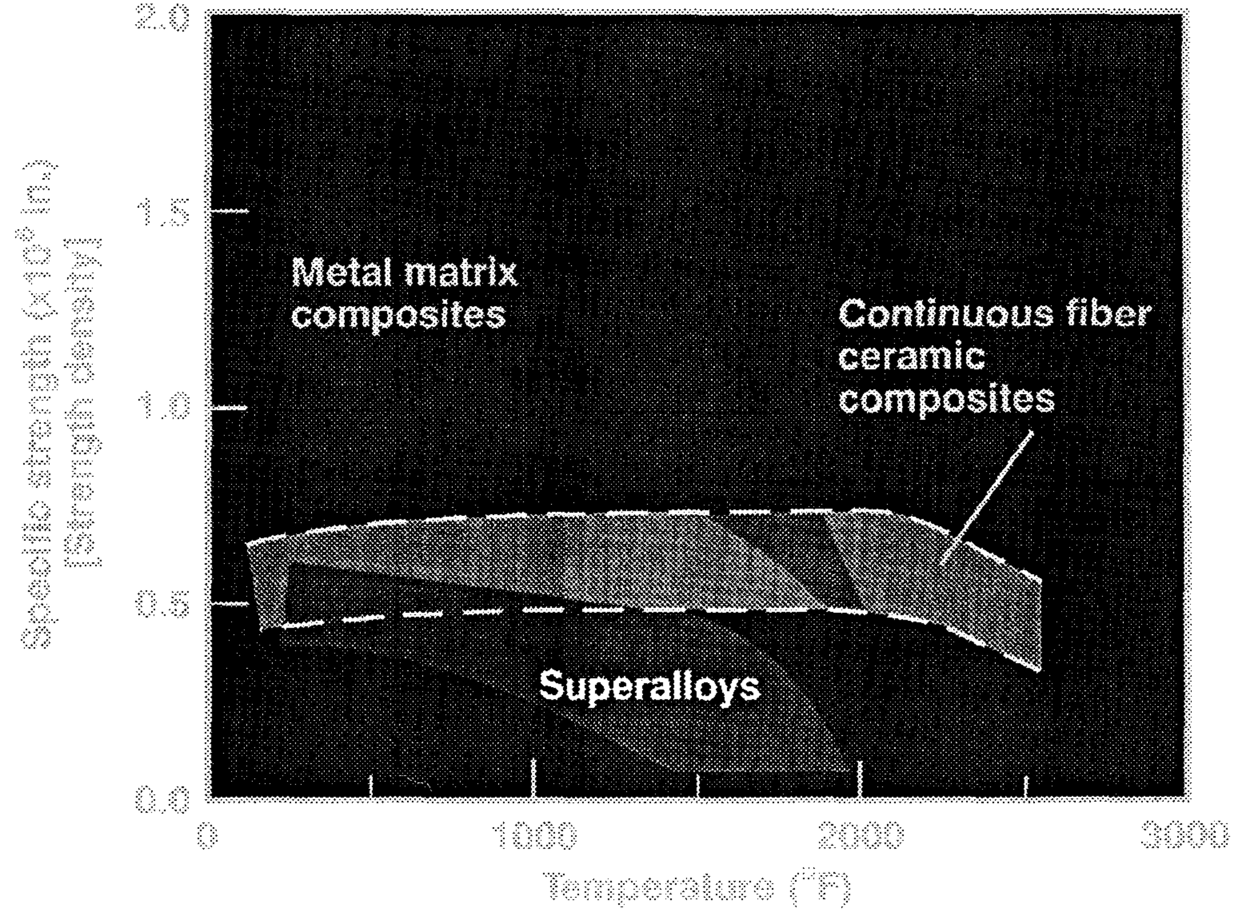
Continuous Fiber Ceramic Composites Retain Strength at Elevated Temperatures



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Potential CFCC Applications in METC Advanced Power Systems

	Ceramic filters	Gas turbine hot path components	Combustion liners	Heat exchangers	Fuels cell structures	High wear components	Operating temperatures
PFBC	●	●	●				1400°F to 2800°F
IGCC	●	●	●				800°F to 2800°F
EFCC			●	●			1500°F to 3000°F
IGFC	●				●		800°F to 1800°F
ATS		●	●				1500°F to 2800°F
Coal fueled diesel						●	
Exposure to contaminants	Volume	Surface	Surface	Surface	Volume		

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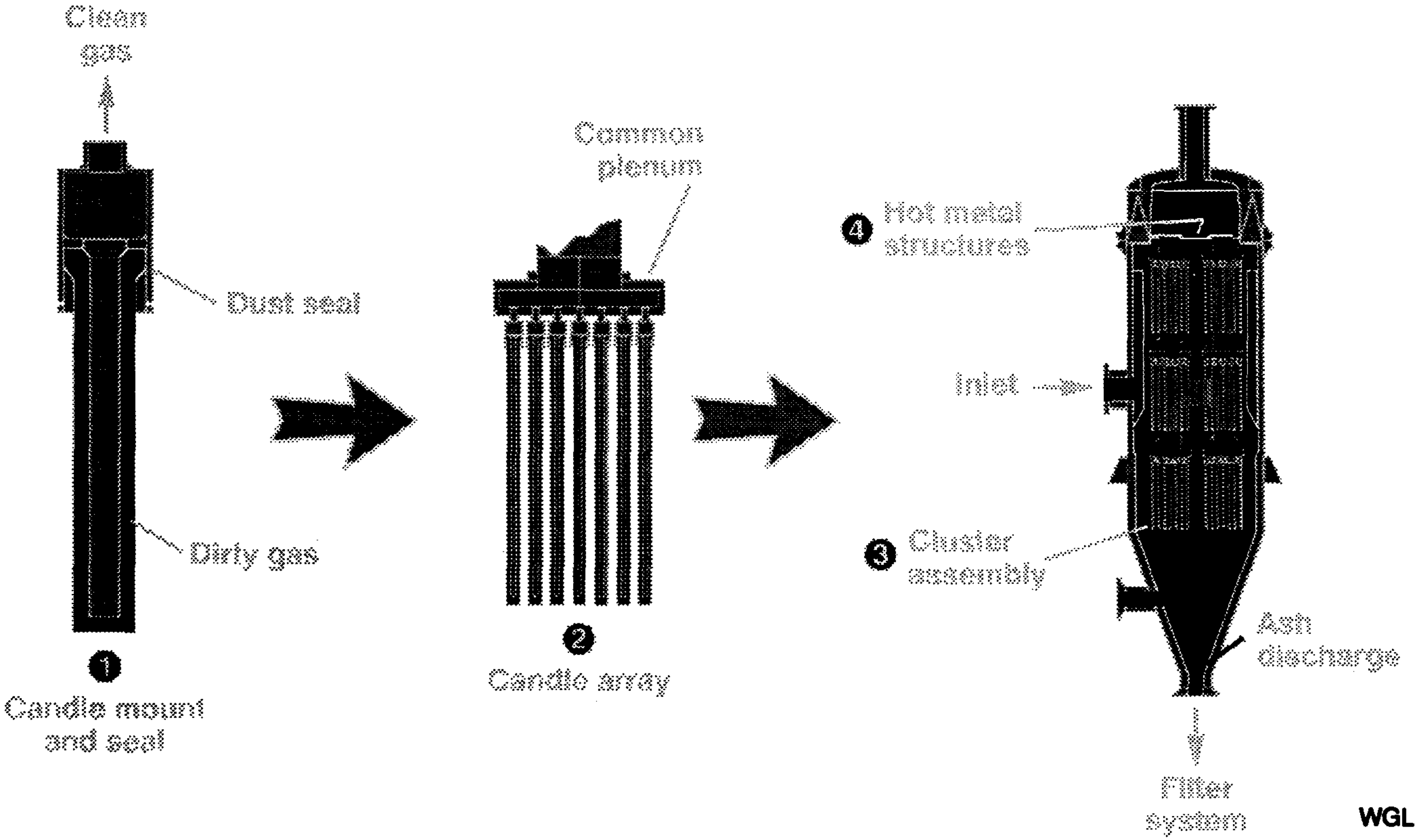
Westinghouse Candle Filter System



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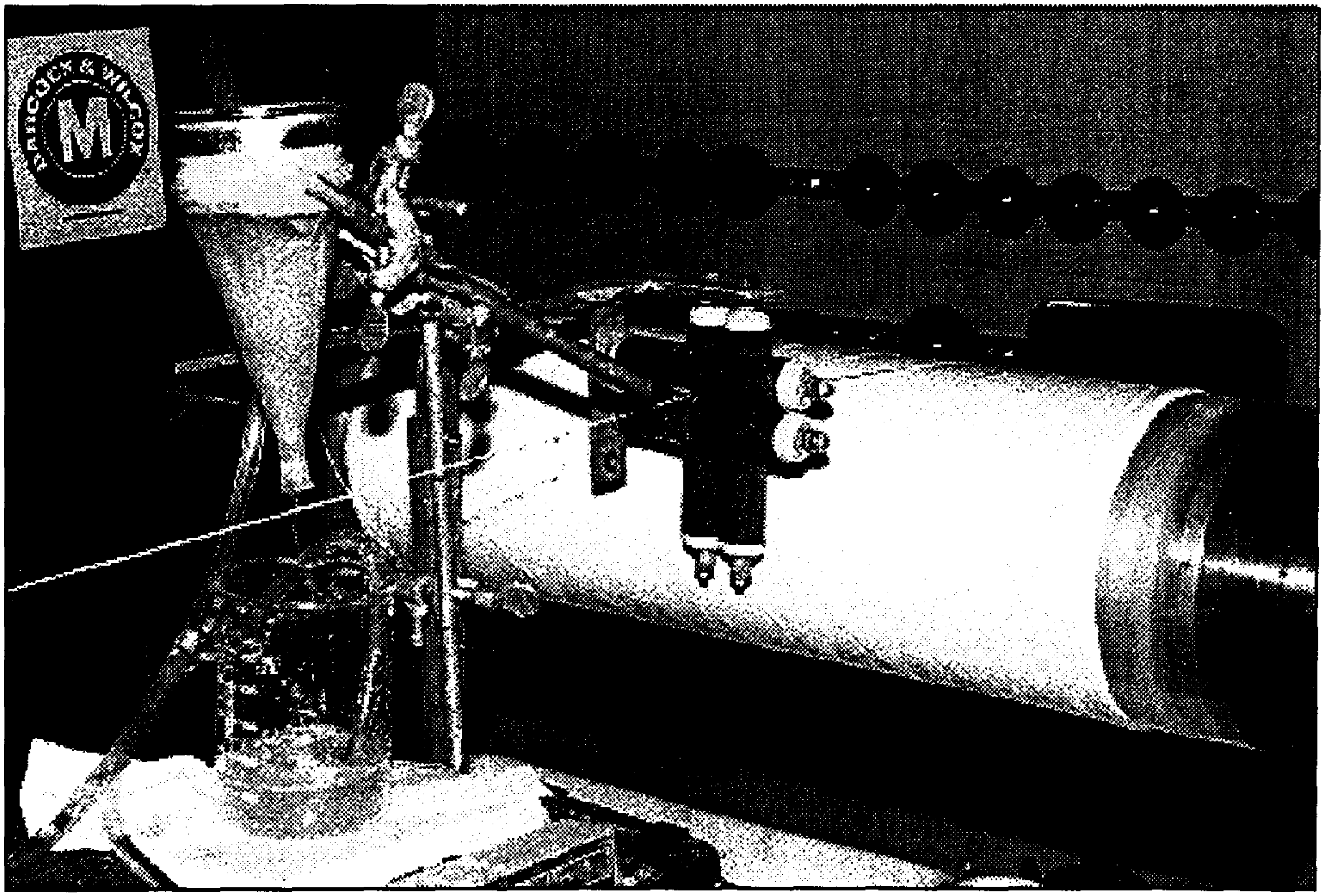
Filament Winding a Turbine Combustor



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DESIGNING WITH CONTINUOUS FIBER CERAMIC COMPOSITES

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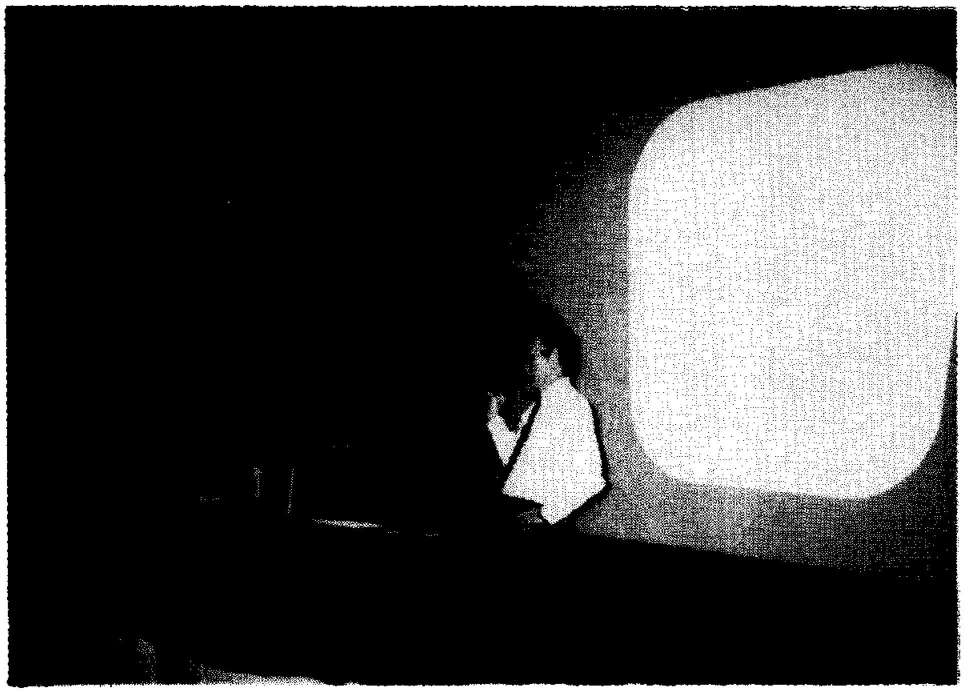
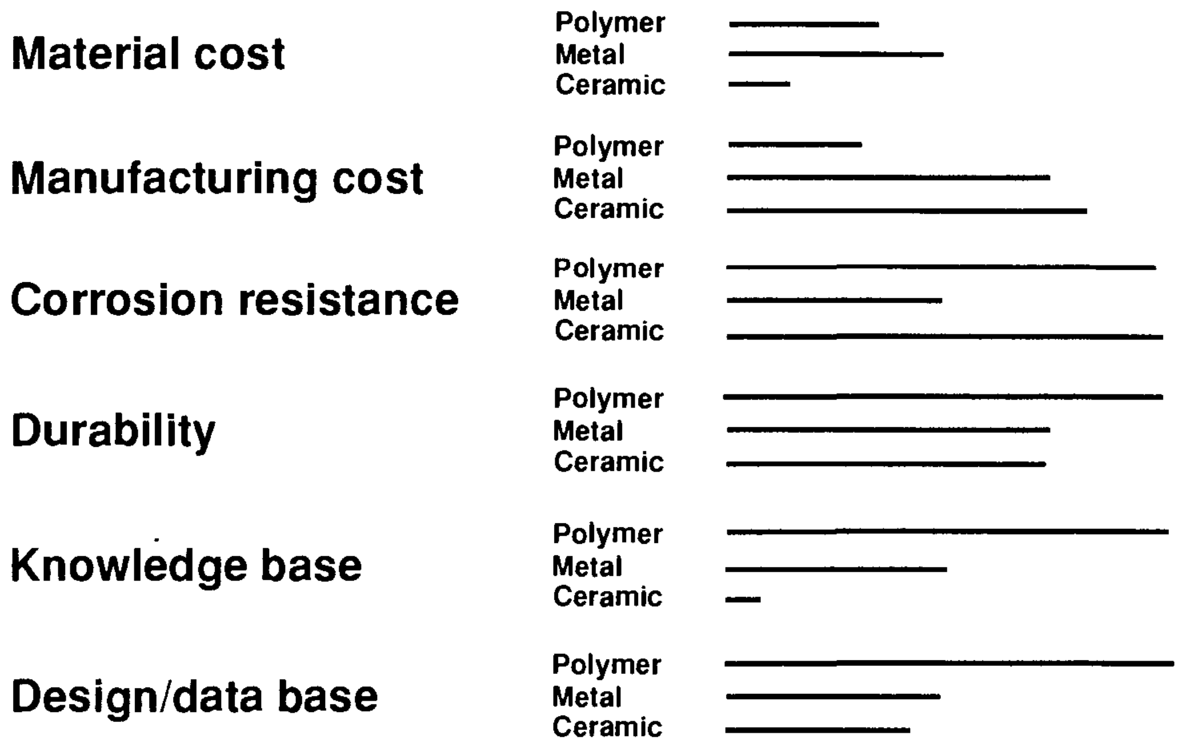


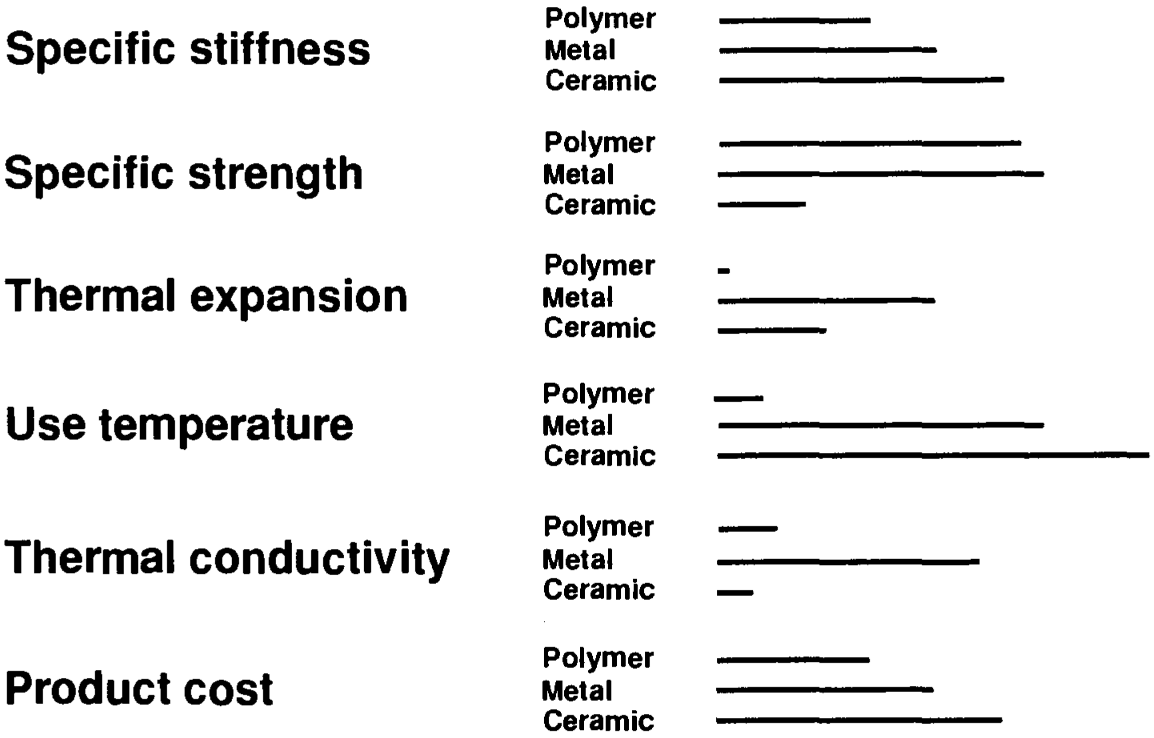
Designing with Continuous Fiber Ceramic Composites

Ken Reifsnider
Materials Response Group, Virginia Polytechnic Institute & State University

Comparative Utility



Comparative Utility



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Ceramic Matrix Composites

Applications -

-> Gas turbines - liners, vanes, shrouds, blades

-> Radiant burners - tubes, combustors

-> Heat exchangers - tubes, headers, manifolds

-> Electronic components - boards, elements, sensors, fiber optic devices

-> Engines - power transfer, exhaust/air transfer systems

-> Hot gas filters - engines, power plants

Ceramic Matrix Composites

Manufacturing industry applications -

-> Steel soaking pits

-> Aluminum remelt furnaces

-> Glass melting furnaces

-> Recycling facilities

Ceramic Matrix Composites

Projected advantages -

-> Engines - 10-35 percent fuel savings

-> Heat exchangers - recovery of large part of
the 25 to 65 percent heat loss generated
by high temperature combustion systems

Ceramic Matrix Composites

-> *Properties and end uses of advanced ceramics*

Material	Flexural strength, MPa[a]	Hardness (Vickers), GPa[b]	Fracture toughness, MPa $m^{1/2}$	Maximum use temperature, °C	Young's modulus[c], GPa	End uses
Alumina	310	17	4	1200	310	Wear parts, cutting tools
Silicon carbide	690	22.4	4	2000	450	Wear parts, cutting tools, heat exchangers
Silicon nitride	925	15.9	5.5	1400	315	Wear parts, automotive engine applications
Zirconia	1440	12.8	8.5	800	220	Cutting tools, wear parts, experimental heat engines
Tool steel	5500	10	98	700	210	Cutting tools, wear parts

[a] Megapascals.
[b] Gigapascals.
[c] Young's modulus defines the ratio between stress and strain and is an indicator of the elasticity of a material.

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Ceramic Matrix Composites

-> Advanced structural composites; applications and markets

Table 3. Various current and potential applications of ASC and application requirements

Industry	Application	Application requirements
Machine tool	Cutting tools Bearings Wire drawing dies	Wear and corrosion resistance, minimum lubrication requirements
Petrochemical	Seals Valves Pump impellers Heat exchangers	Energy-efficient heat regeneration
Automotive	Turbocharger rotors Push rod tips Rocker arms Cylinder liners	Light-weight, high-temperature, corrosion and wear resistance
Defense	Gun liners Ceramic armor	Light-weight, strength, corrosion, and high-temperature resistance

Table 2. Estimated size of current and projected U.S. markets for ASC (in millions of dollars) *(6)*

Item	1992	2000
Wear parts	120	540
Cutting tool inserts	85	300
Bearings	75	300
Bioceramics	20	60
Heat exchangers	20	100
Automotive/heat engine	0	920
Aerospace, defense	80	450

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Ceramic Matrix Composites

-> *Projected development of ceramic components*

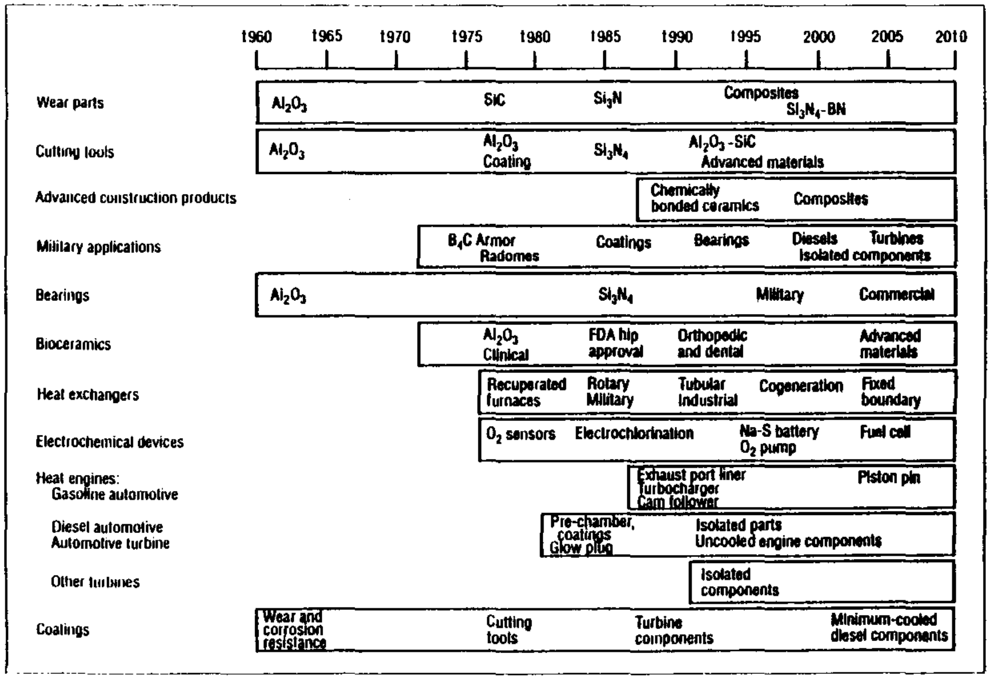

Figure 1. **Estimates for implementation of ceramic components in structural application categories *(4).***

46 CHEMTECH NOVEMBER 1993

Ceramic Matrix Composites

-> Application examples - benefits

DOW CORNING

Task 1 Applications Assessment
Solar Turbines - Benefits

Energy Savings - Fleet of Metallic and Ceramic Engines to Meet Predicted Needs

Year	Cumulative GW Installed	Engines Required to Satisfy Need	
		Metallic	Ceramic
2005	8	814	571
2010	24	2443	1714
2015	40	4071	2856

Environmental Savings - Comparative NOx Emissions in Tons/Year for Metallic vs. Ceramic Engine

Year	GW Demand (Cumulative)	Metallic Mars T14000		Ceramic Mars	
		Need	Emissions	Need	Emissions
2005	8	814	153	571	8.6
2010	24	2443	459	1714	25.9
2015	40	4071	765	2856	43.1

Strategic Elements Savings/Engine: Cr, Co, W, Mo, Cb, Ni

Component	Total Unfinished Weight (lbs.)
Combustor	447
Turbine	1408
Turbine Diffuser Housing	839
Total	2694

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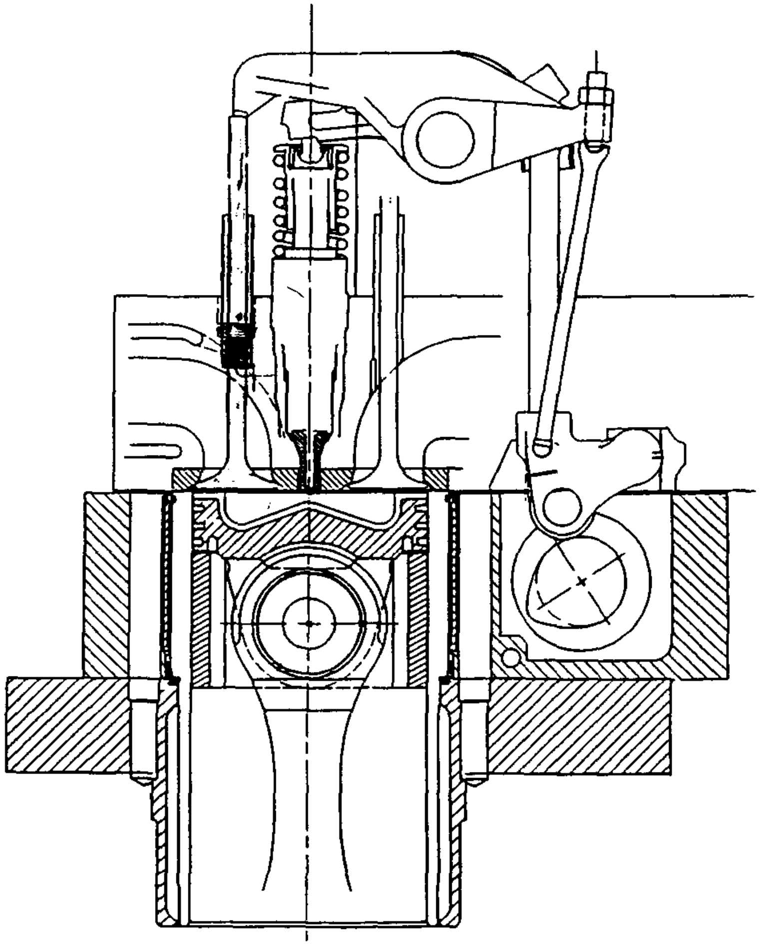
Ceramic Matrix Composites

-> Application example - valve guides

CATERPILLAR

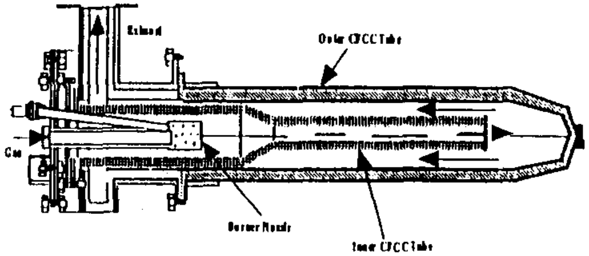


Ceramic Matrix Composites

-> Application example - immersion heater tubes

PROSPECTIVE CFCC COMPONENT APPLICATIONS

- *APPLICATION AREA:* PRIMARY AND SECONDARY METALS PROCESSING
- *PROSPECTIVE CFCC COMPONENTS:* RADIANT OR IMMERSION HEATER TUBES

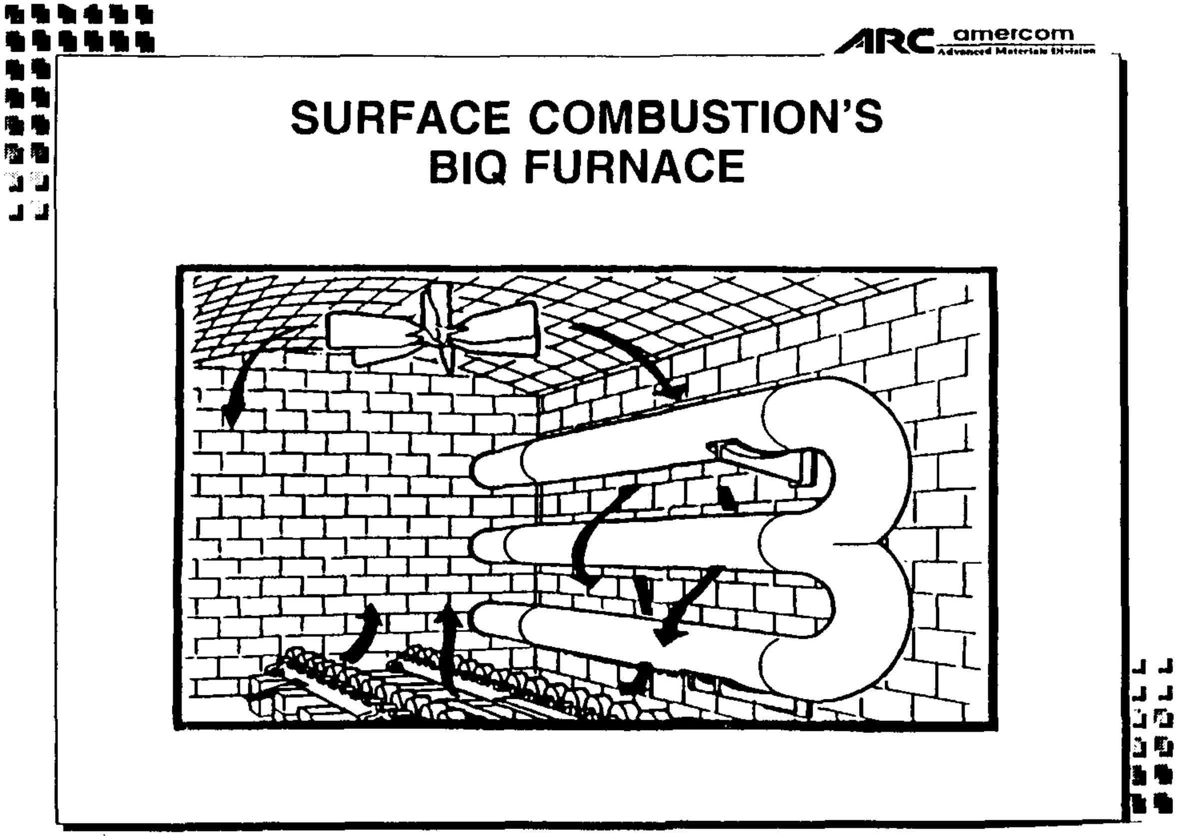


TEXTRON
Specialty Materials

- *TYPICAL ENVIRONMENTS:*
  - OXIDIZING, MOLTEN METAL OR INERT
  - 2300-2600°F (1200-1400°C) OPERATING TEMPS
  - PRINCIPALLY THERMOMECHANICAL (AND IMPACT) LOADING; (PERHAPS 15-20 ksi (100-130 MPa) STRESS LEVELS)
  - ONE TO FOUR YEAR LIFETIME

Ceramic Matrix Composites

-> *Application example - soaking furnaces*



ARC amercom
Advanced Materials Division

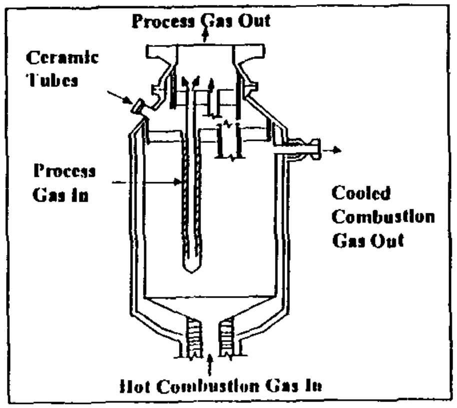
SURFACE COMBUSTION'S
BIQ FURNACE

Ceramic Matrix Composites

-> *Application example - chemical processing*

PROSPECTIVE CFCC COMPONENT APPLICATIONS

- *APPLICATION AREA*: PETROCHEMICAL PROCESSING INDUSTRY
- *PROSPECTIVE CFCC COMPONENT*: CATALYTIC REFORMER OR PYROLYSIS TUBES



TYPICAL ENVIRONMENTS:

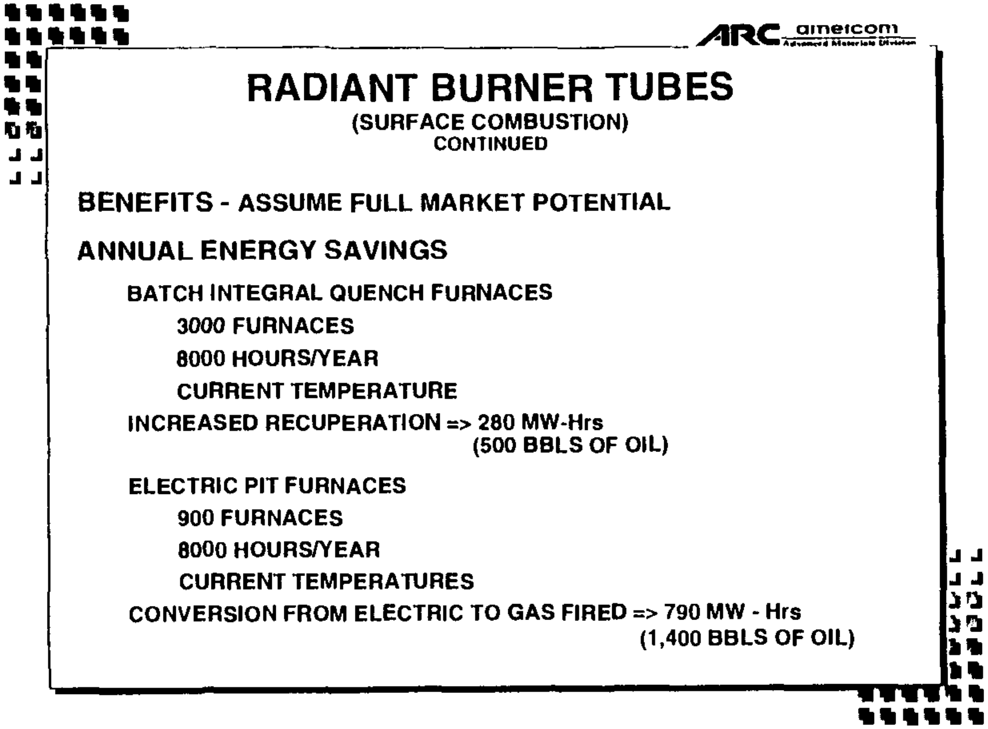
- OXIDIZING; MODERATE-HIGH PARTIAL PRESSURES OF STEAM
- 2300-2600°C) OPERATING TEMPS
- PRINCIPALLY THERMOMECHANICAL (AND IMPACT) LOADING; (PERHAPS 15-20 ksi (100-130 MPa) STRESS LEVELS)
- LIFETIME UP TO TEN OR MORE YEARS

TEXTRON
Specialty Materials

Materials Response Group, Virginia Polytechnic Institute & State University

Ceramic Matrix Composites

-> *Application example - radiant burner tubes*



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RADIANT BURNER TUBES

(SURFACE COMBUSTION)
CONTINUED

BENEFITS - ASSUME FULL MARKET POTENTIAL

ANNUAL ENERGY SAVINGS

- BATCH INTEGRAL QUENCH FURNACES
  - 3000 FURNACES
  - 8000 HOURS/YEAR
  - CURRENT TEMPERATURE
- INCREASED RECUPERATION => 280 MW-Hrs (500 BBLS OF OIL)
- ELECTRIC PIT FURNACES
  - 900 FURNACES
  - 8000 HOURS/YEAR
  - CURRENT TEMPERATURES
- CONVERSION FROM ELECTRIC TO GAS FIRED => 790 MW - Hrs (1,400 BBLS OF OIL)

Ceramic Matrix Composites

Technical Constraints -

-> Production methods and costs

-> Reliability, reproducability, inspection methods

-> Availability of design data (static, long term)

-> Thermal stability

-> Supporting technologies -

- Characterization methodologies
- Durability, damage tolerance, life prediction
- Design methodologies

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Ceramic Matrix Composites

Preliminary Design Properties -

-> Stiffness and strength (all anisotropic values)

-> Coefficients of thermal expansion

-> Thermal conductivity

-> Thermal diffusivity

-> Creep, creep-rupture, fatigue behavior

-> Environmental degradation

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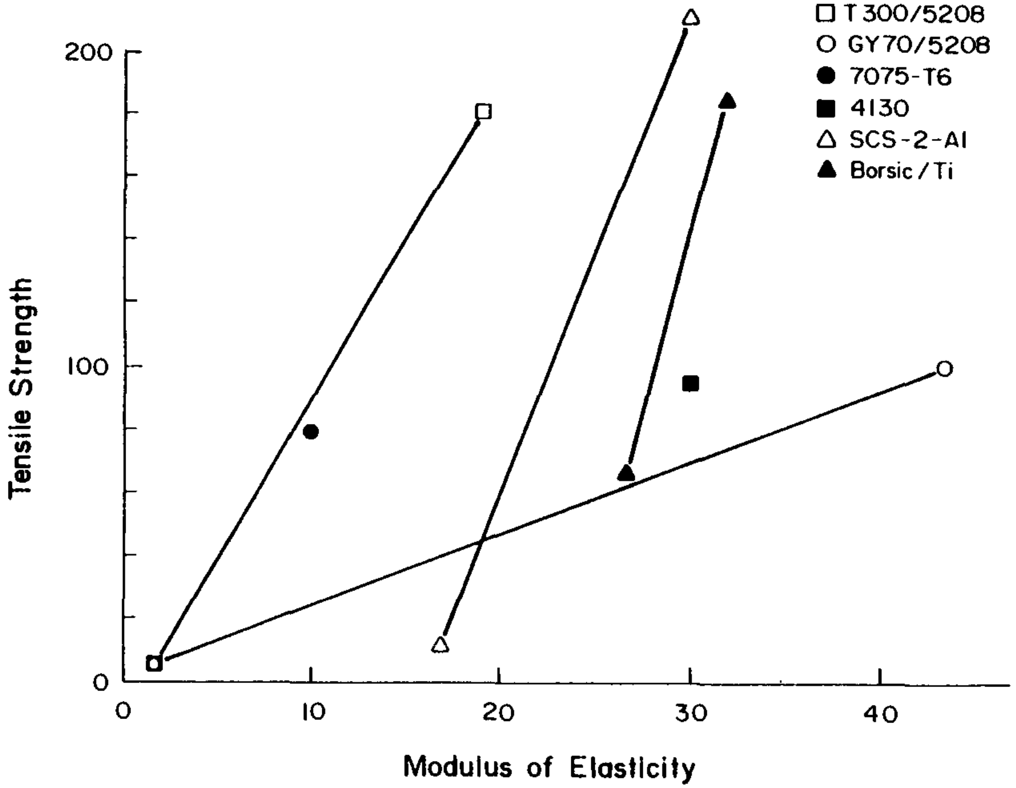
Ceramic Matrix Composites

-> **Each composite system offers a range of properties and performance**



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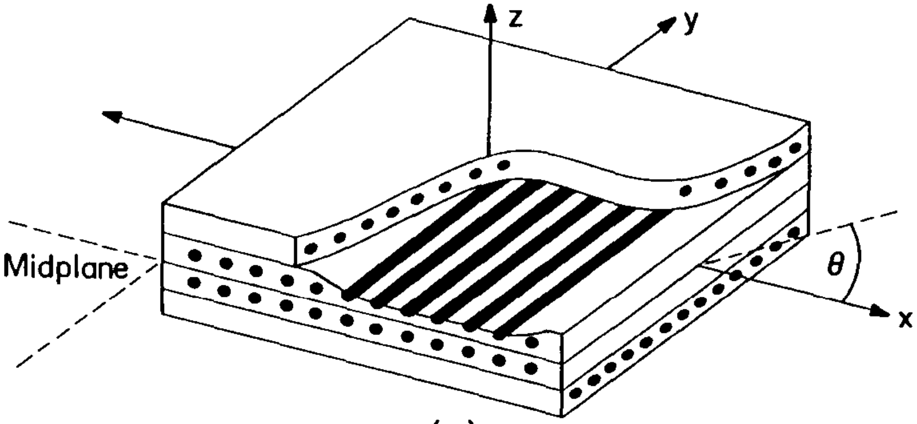
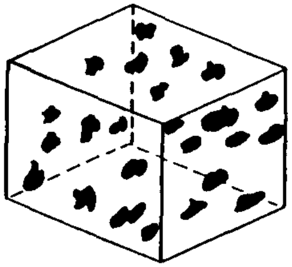
Ceramic Matrix Composites

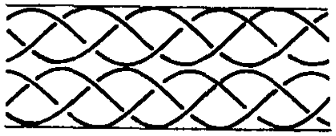
-> **Reinforcement is the key to properties and performance**



(a)



(b)



(c)

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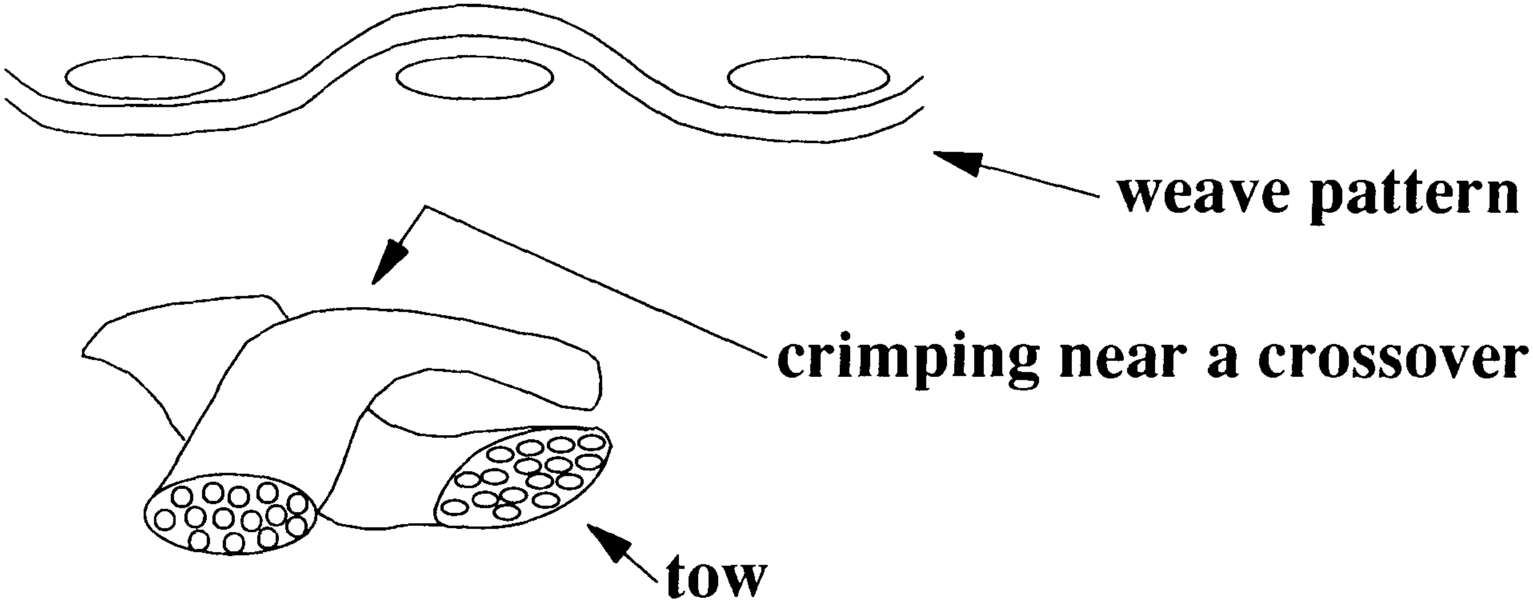
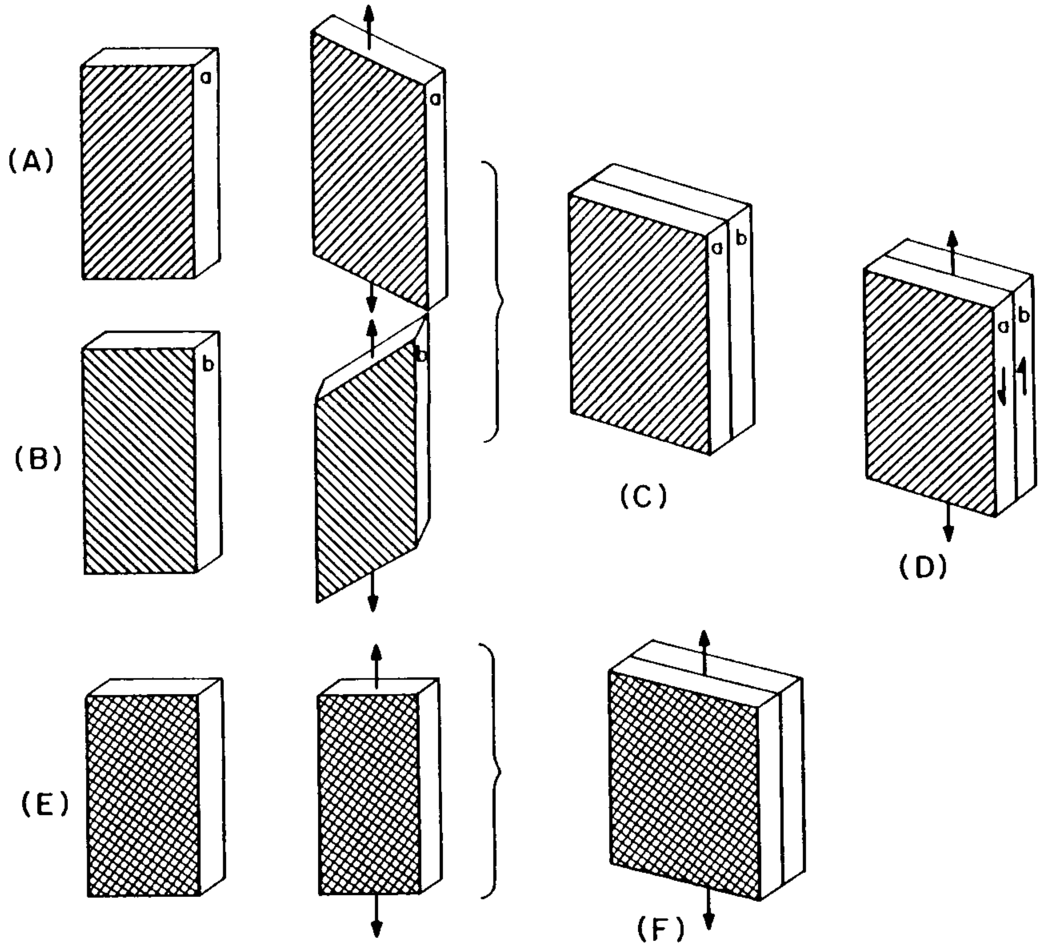
Ceramic Matrix Composites

-> ***Local fiber architecture influences performance***



Ceramic Matrix Composites

-> Internal constraints cause thermal stresses



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Ceramic Matrix Composites

Durability -

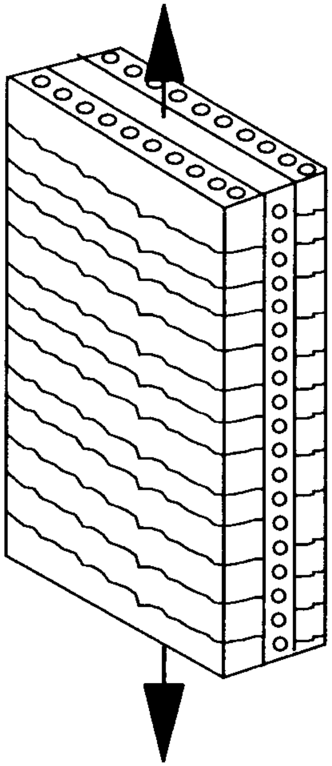
-> Damage tolerance (fatigue)

-> Resistance to creep, creep rupture

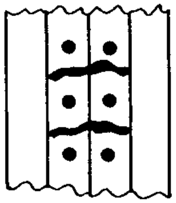
-> Resistance to aging
  - chemical
  - physical

Ceramic Matrix Composites

-> *Matrix cracking is a dominant damage mode*

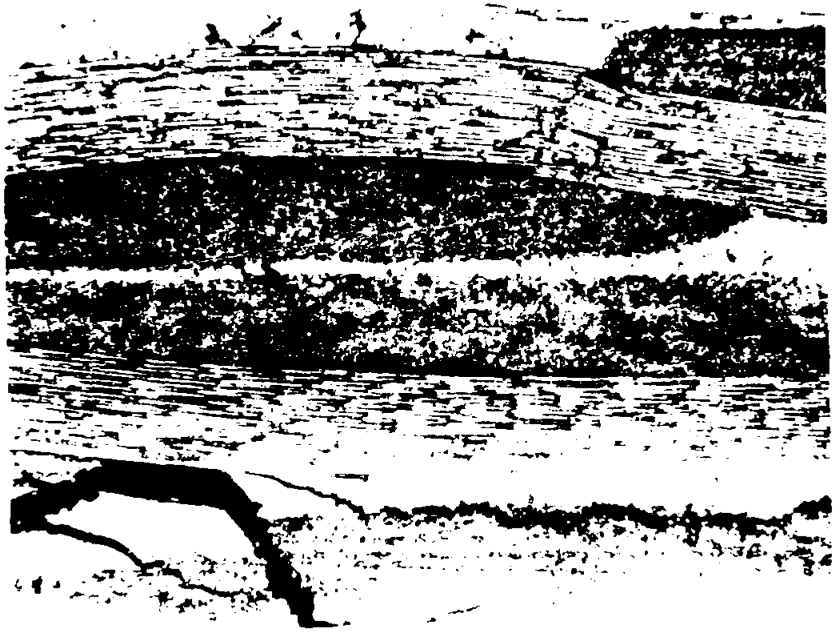


Fibers control the effect of matrix cracking



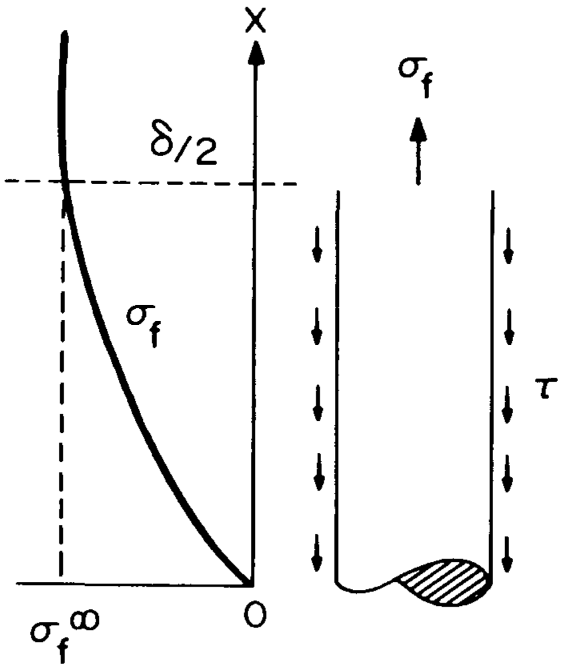
Ceramic Matrix Composites

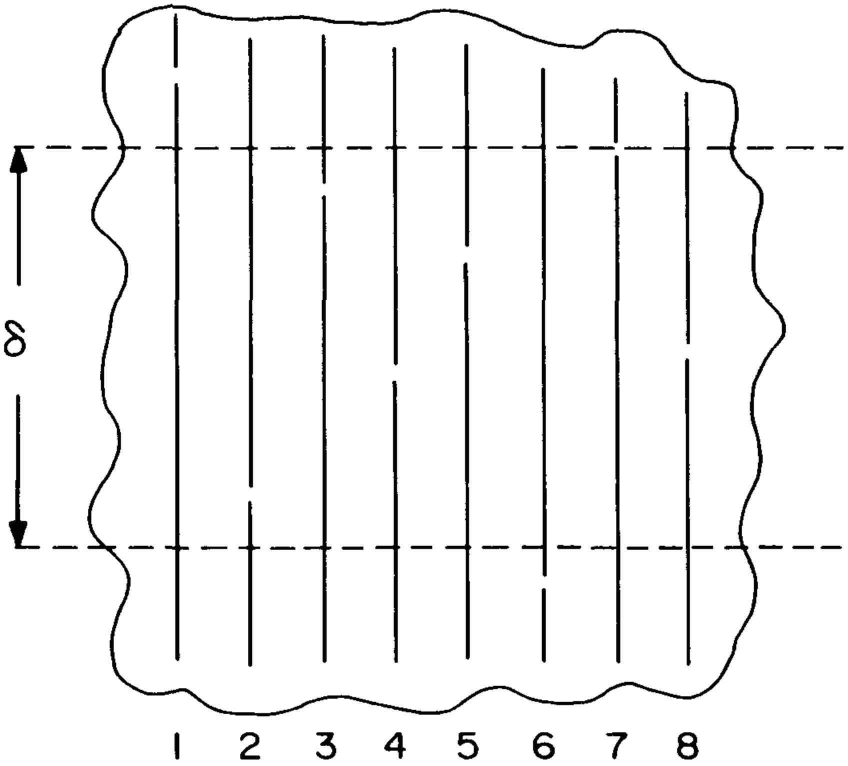
-> *Local damage modes control damage tolerance*



Ceramic Matrix Composites

-> *Fibers can break many times before "rupture"*





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Ceramic Matrix Composites

-> *Damage tolerance (fatigue)*

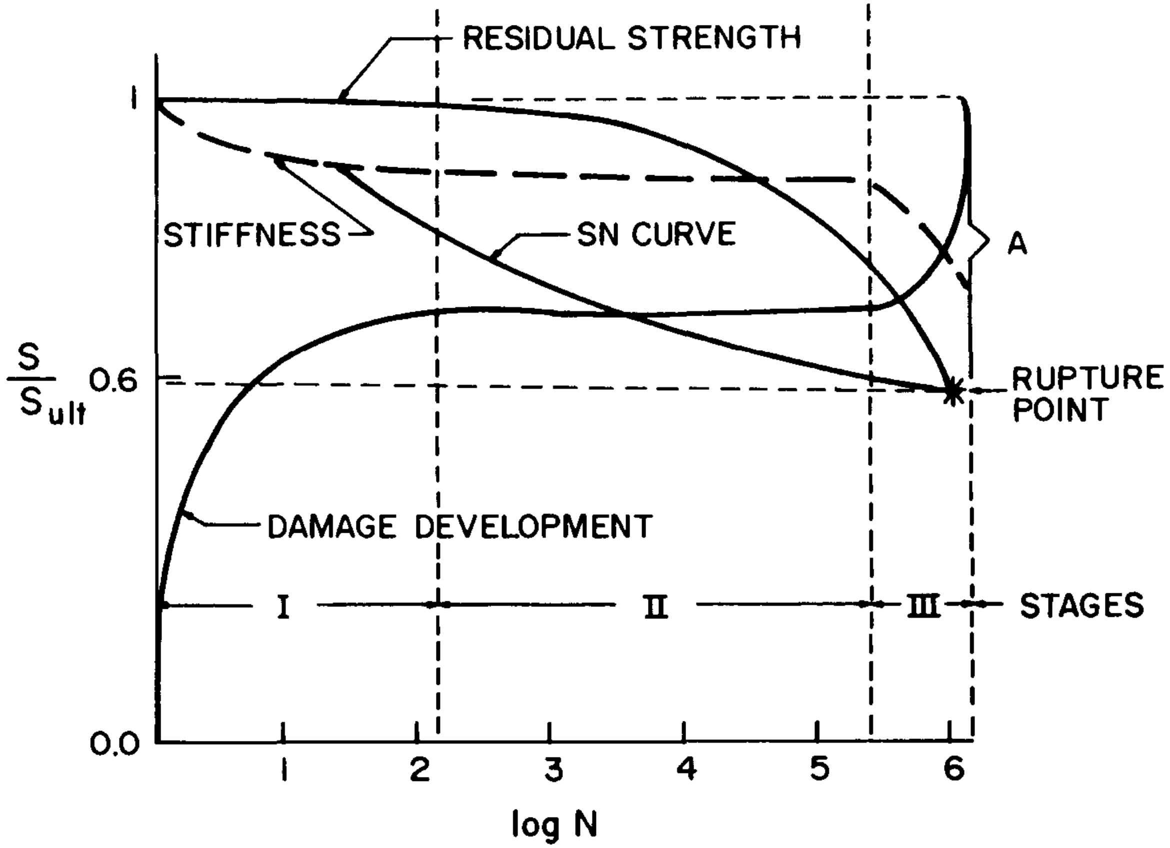
* Failure modes are similar
to those for short-term behavior

* Internal stress redistribution
results from damage modes

* Composite systems fail from the
(statistical) accululation of defects

Ceramic Matrix Composites

-> Damage develops by accumulation



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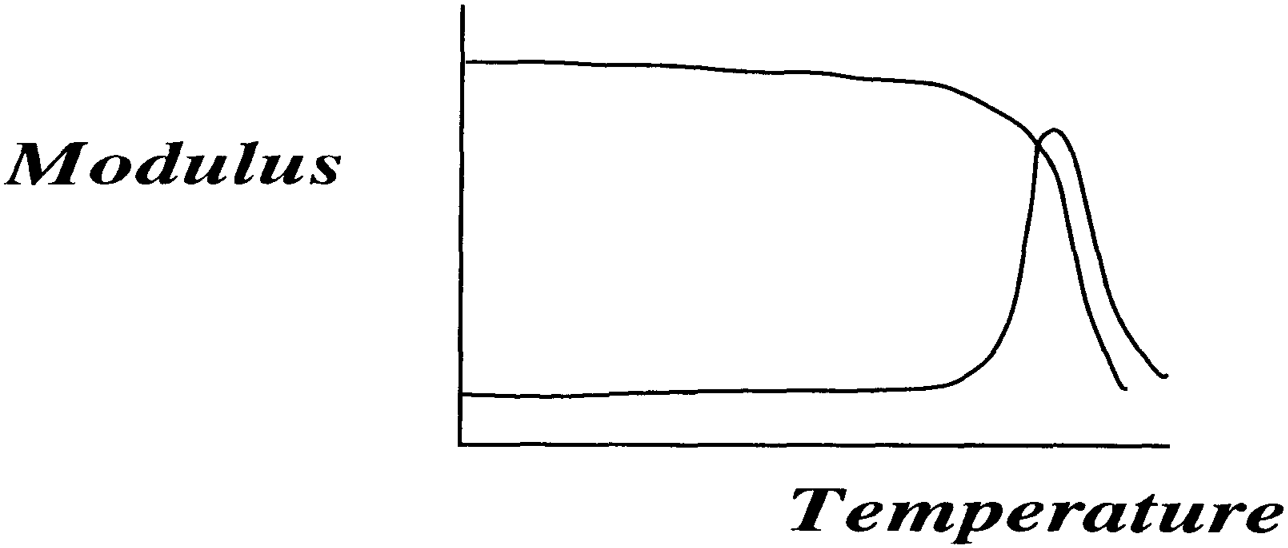
Ceramic Matrix Composites

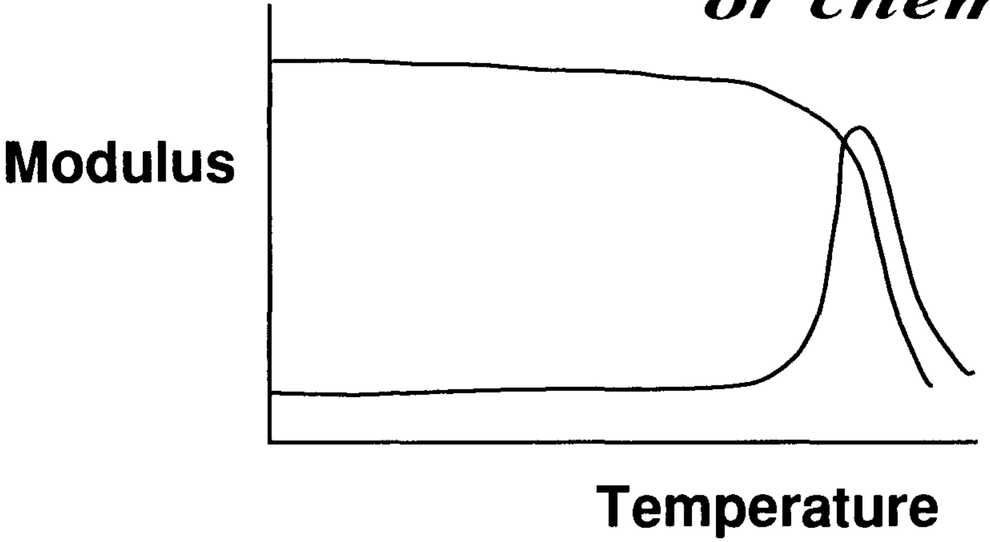
-> *Resistance to (physical, chemical) aging*



Ceramic Matrix Composites

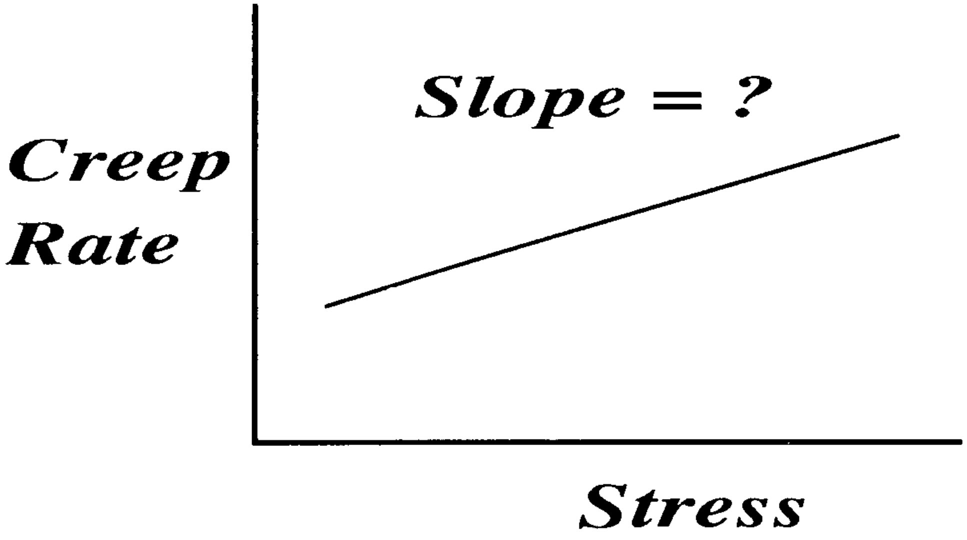
-> *Resistance to (physical, chemical) aging*

* *Chemical reaction rates or diffusion may control rate or chemical aging*



Ceramic Matrix Composites

-> Resistance to creep, creep rupture



Ceramic Matrix Composites

-> *Resistance to creep, creep rupture*

* Combined effects may be "anomalous"

* Mechanisms control the
slope of the rate curve

* Temperature greatly affects
the controlling mechanism

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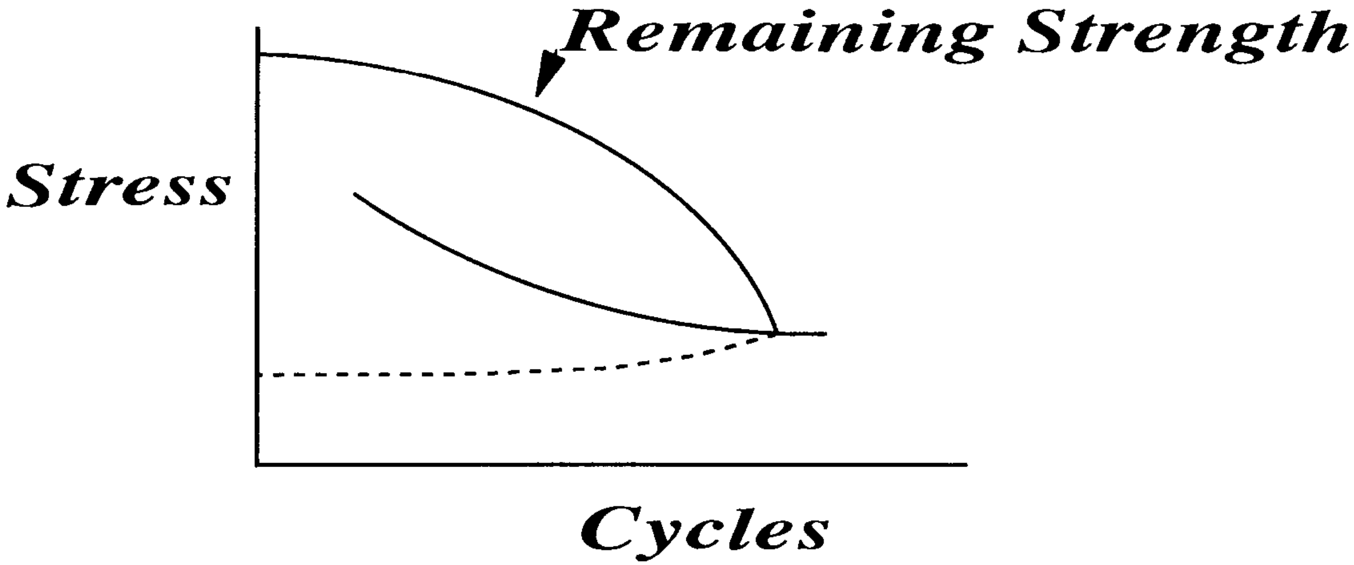
Ceramic Matrix Composites

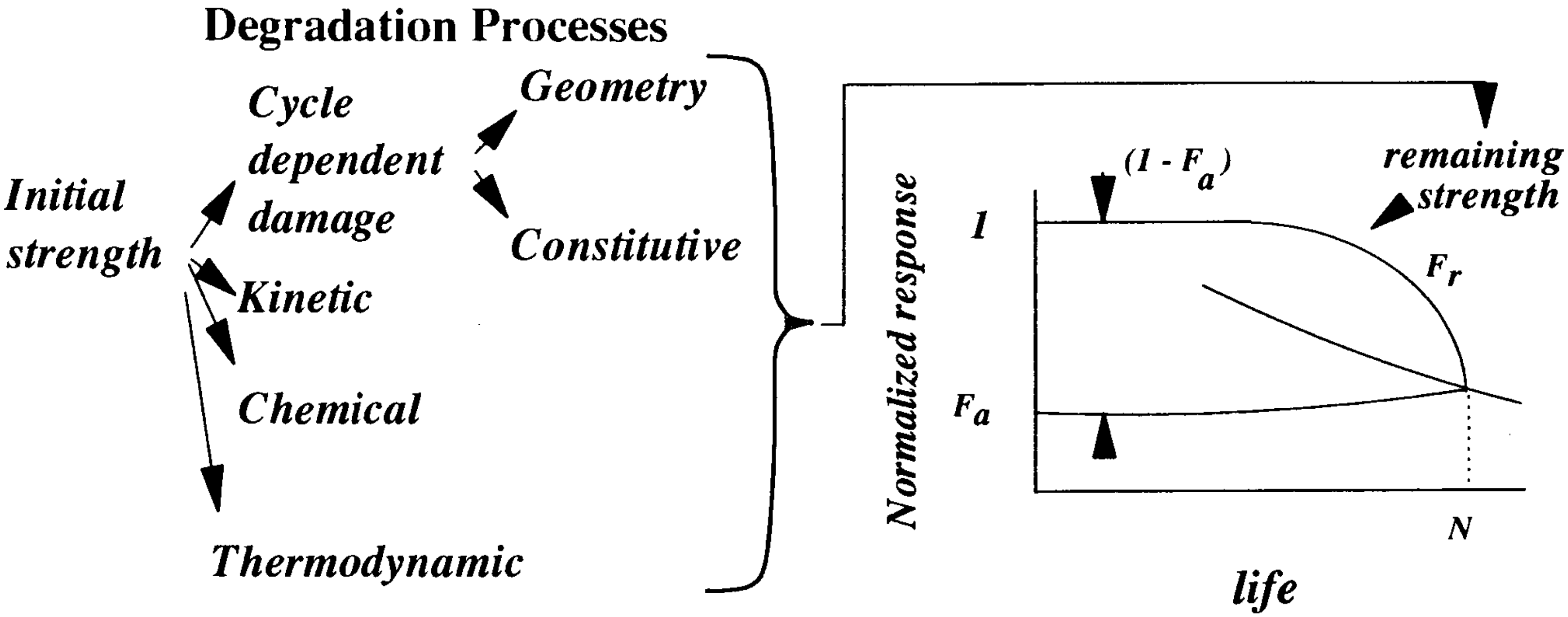
-> *Damage tolerance (fatigue)*



Ceramic Matrix Composites

Durability - The Problem:

How can we combine our data and understandings of material system behavior under long-term conditions to estimate remaining strength and life, or to make a better material system?



Degradation Processes

Initial strength

Cycle dependent damage

Geometry

Constitutive

Kinetic

Chemical

Thermodynamic

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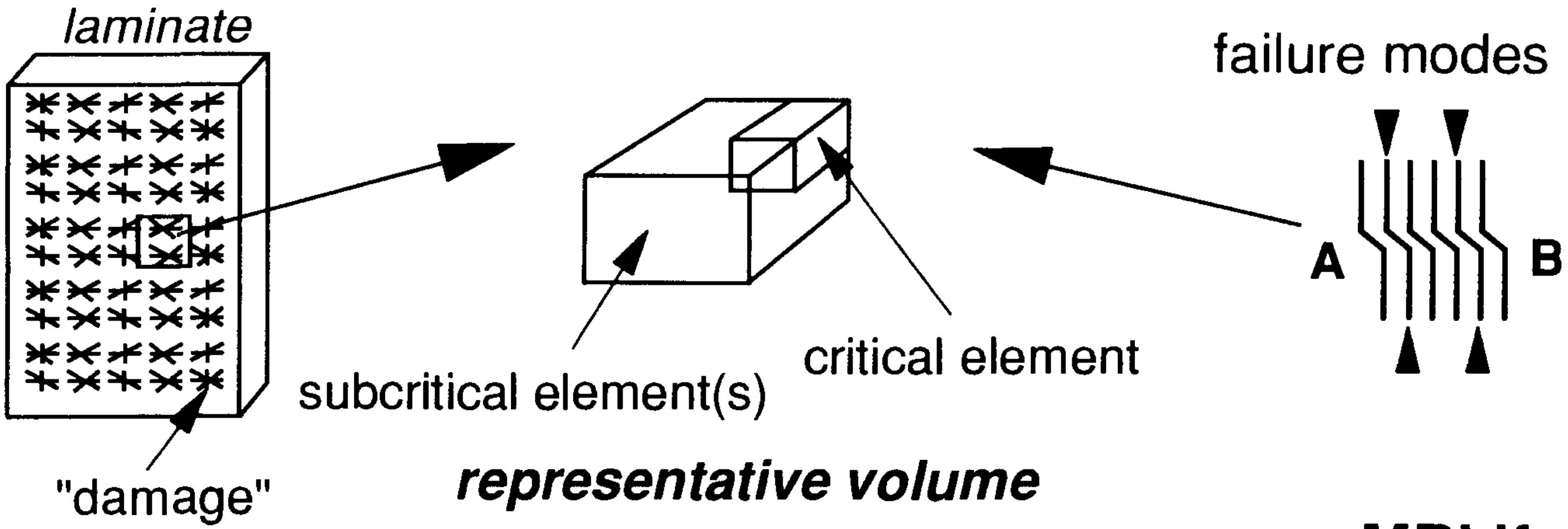
Ceramic Matrix Composites

Our Approach:

"Critical Element Concept"

- identify a local material element whose failure defines global failure for a given failure mode.



MRLife ™

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Ceramic Matrix Composites

Degradation Processes Modeled:

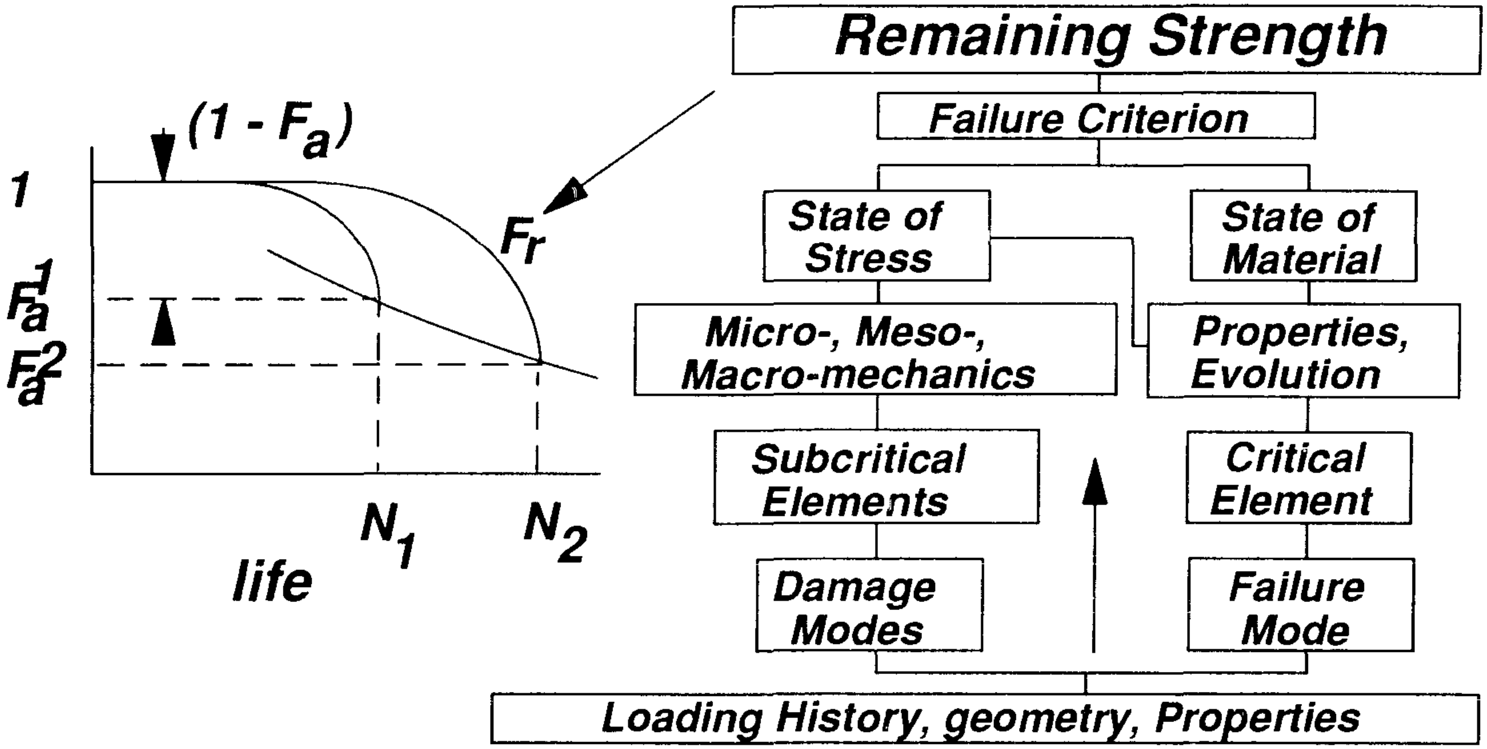
Phenomena	Stiffness Change	Strength Change
Stress relaxation	X	
Oxidation		X
Viscoelastic creep	X	
Creep rupture		X
Moisture effect	X	X
Aging	X	X
Environmental degradation		X
Temperature effect	X	X

MRLife ™

Materials Response Group, Virginia Polytechnic Institute & State University

Ceramic Matrix Composites

MRLife Approach:



Remaining Strength

Failure Criterion

State of Stress

State of Material

Micro-, Meso-, Macro-mechanics

Properties, Evolution

Subcritical Elements

Critical Element

Damage Modes

Failure Mode

Loading History, geometry, Properties

Ceramic Matrix Composites

Residual Strength Calculation:

$$F_r(n/N) = \left[1 - \int_0^{(n/N)_1} (1 - F_a(n/N))\, i(n/N)^{i-1}\, d(n/N) \right]$$

Residual strength

Failure criterion

1. K / K_c
2. Tsai-Hill
3. $\Delta E / \Delta E(critical)$
4. (Others...)

Current life

1. Constituent SN
2. Kinetic process life

For a given failure mode

MRLife ™

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Ceramic Matrix Composites

Rate Equations Needed:

>> Chemical - activation rates

>> Cracking - density vs. applied conditions

>> Creep - compliance vs. time (tensor components?)

>> Delamination - rate as a function of energy release

>> Geometry or property degradation rates

Ceramic Matrix Composites

MRLife TM

A Performance Simulation Code for Material Systems

An Opportunity:

The MRLife code, and the critical element philosophy behind it, present a unique opportunity to design for durability, damage tolerance, reliability, and safety in a rigorous, economical, convenient, and competitive manner.

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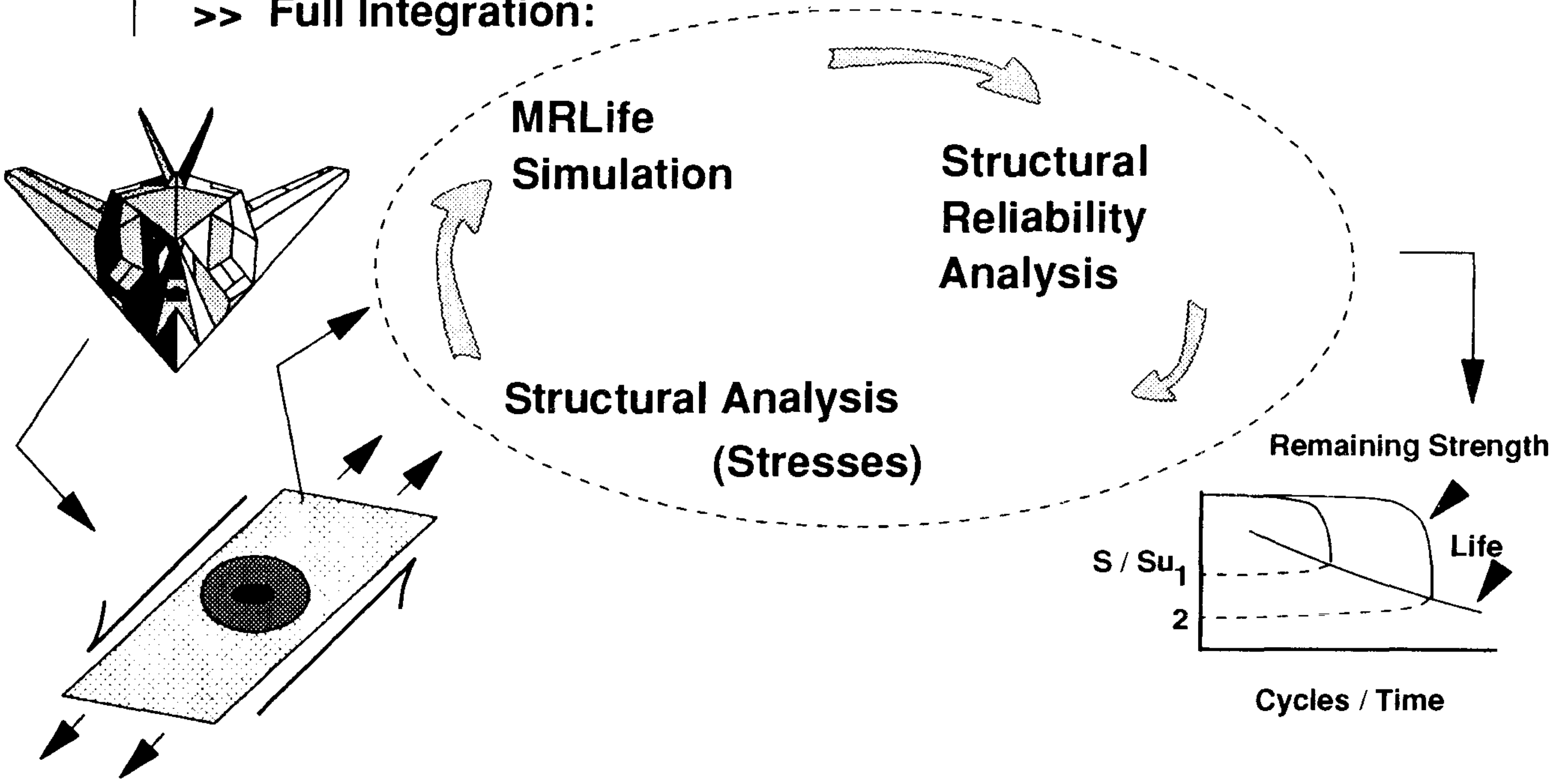
Ceramic Matrix Composites

Implementation:

Integration into Design Codes - MRLife ™

>> Full Integration:



MRLife Simulation

Structural Reliability Analysis

Structural Analysis (Stresses)

Remaining Strength

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Designing with Continuous Fiber Ceramic Composites (CFCCs)

Summary:

- CFCCs have unique high temperature and aggressive environment capabilities
- We can design safe, reliable structures with CFCCs
- More experimental characterization and design experience will make the design effort quicker and easier

Ken Reifsnider
Materials Response Group, Virginia Polytechnic Institute & State University

Ceramic Matrix Composites

Other issues -

-> Joining, load application methods

-> Process control methods, methodologies

-> Processing / property relationships

-> Basic understanding of behavior

NANOSTRUCTURED MATERIALS

Robert D. Shull

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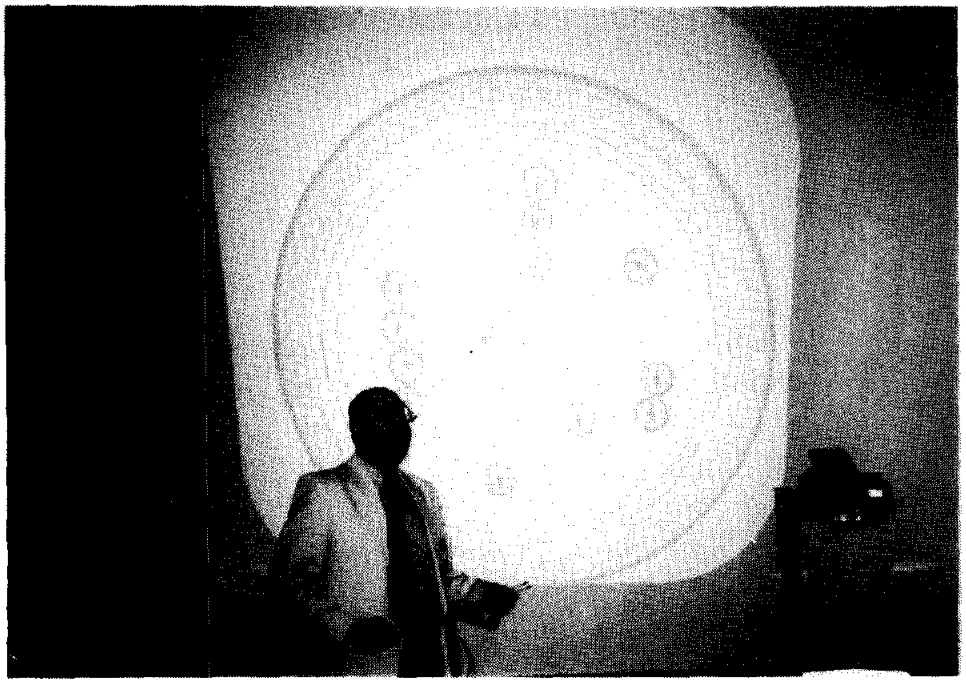
B-140, Materials
Gaithersburg, Maryland 20899

Telephone

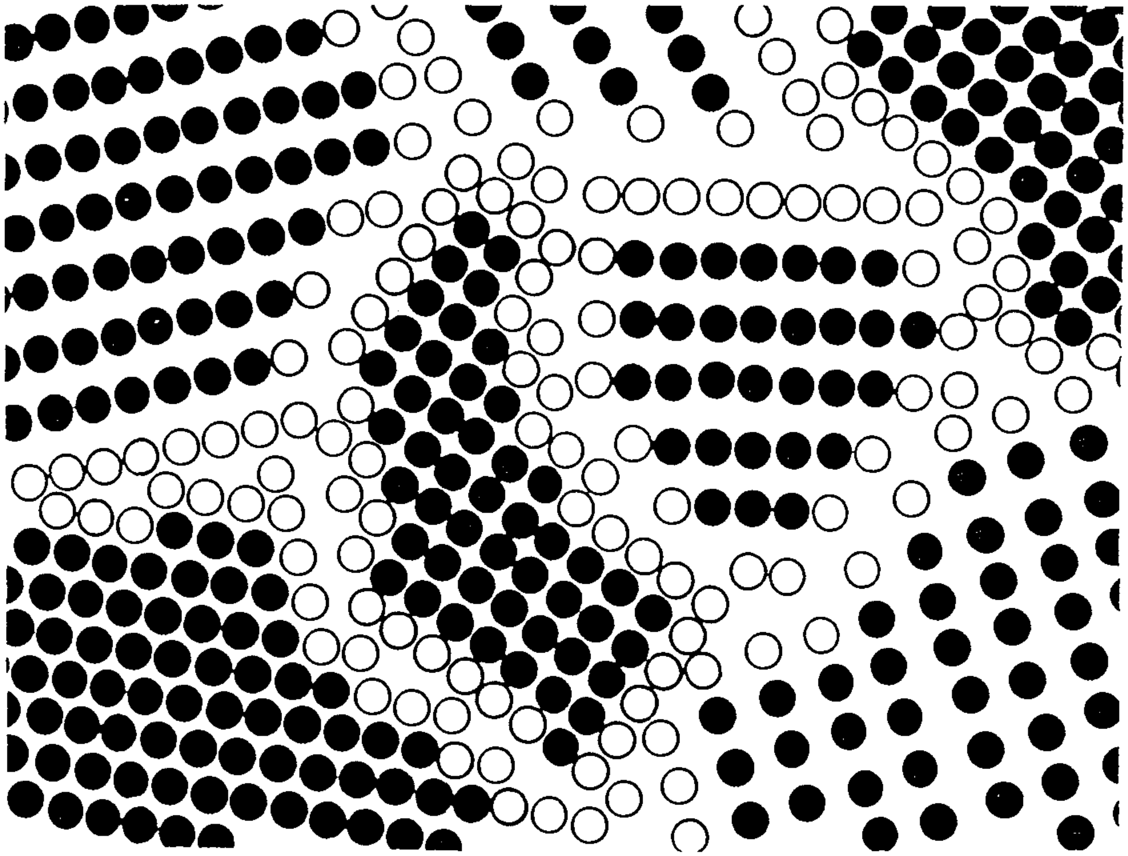


NANOSTRUCTURED MATERIALS

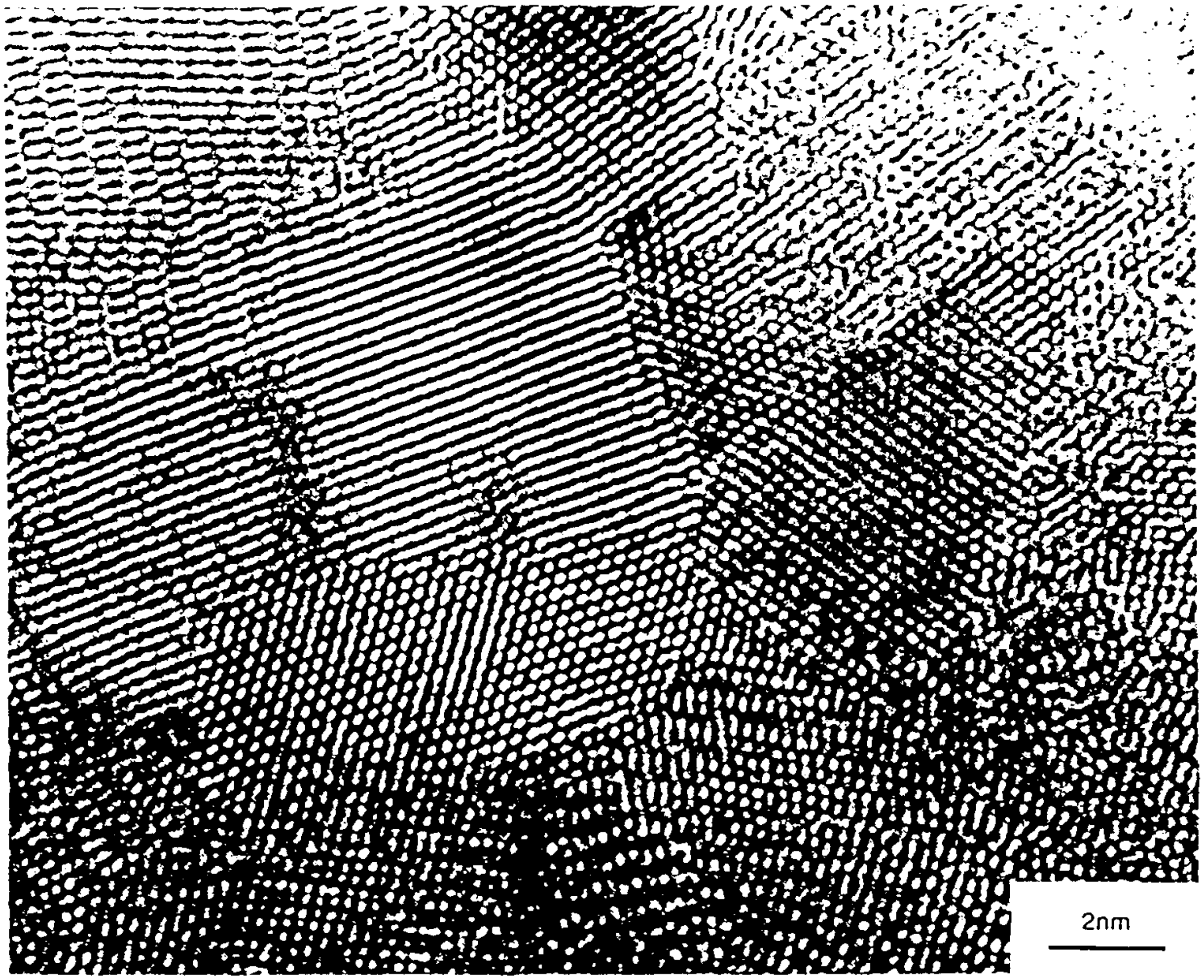
Robert D. Shull

(Magnetic Materials Group)
Metallurgy Division
National Institute
of Standards and Technology
Gaithersburg, Maryland
USA

STRUCTURE SCHEMATIC:
-NANOPHASE MATERIAL-



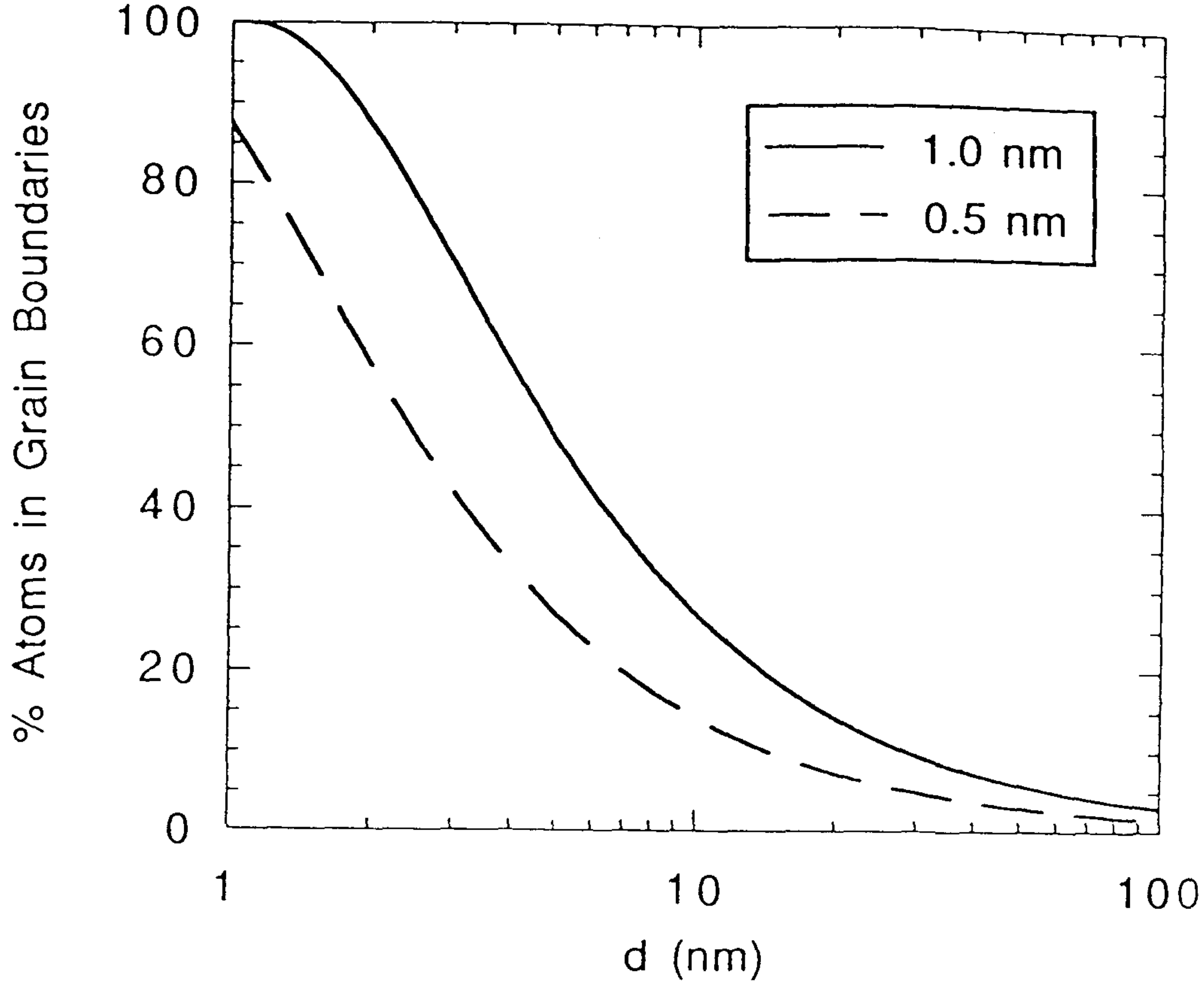
Schematic of an equiaxed nanocrystalline metal showing atoms associated with individual grains (filled circles) and those constituting the grain boundaries (open circles). [H. Gleiter, Prog. Mater. Sci. 89, 223 (1989)]



High resolution TEM picture of Nanophase Pd.

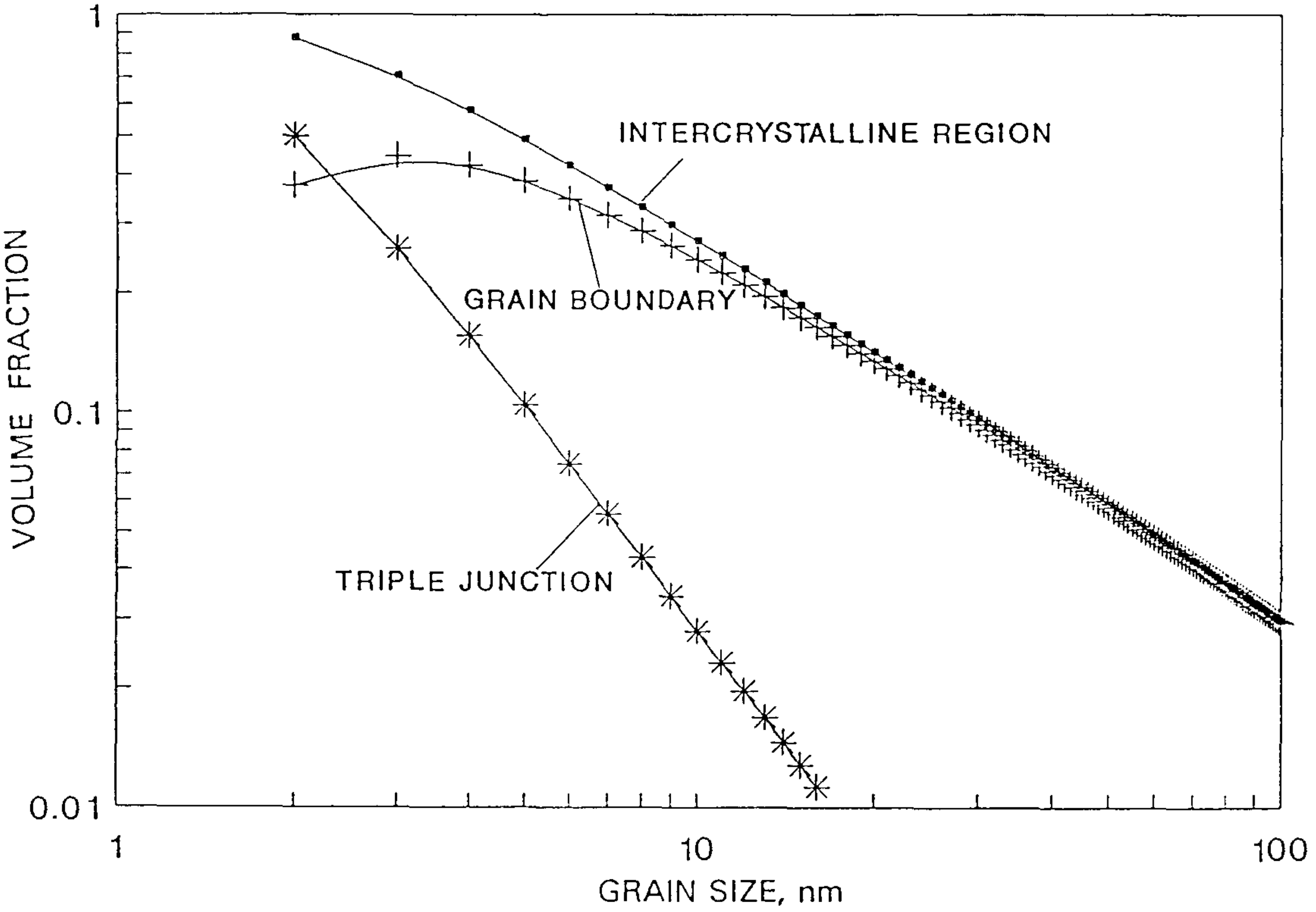
[G.J. Thomas, R.W. Siegel, and J.A. Eastman, Scri. Metall. 24, 201 (1990)]

HIGH INTERFACE VOLUME
-NANOPHASE MATERIALS-



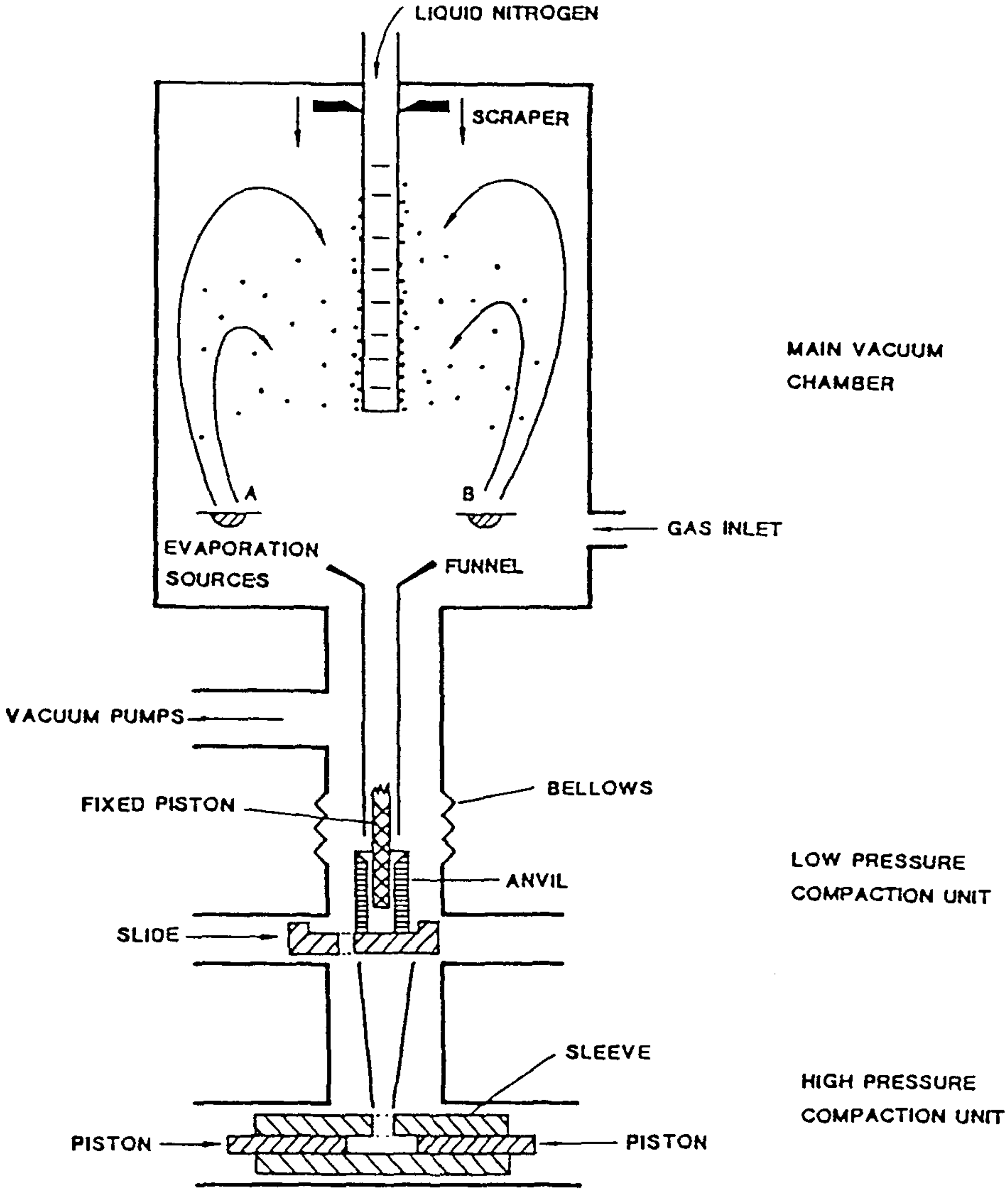
Percentage of atoms in grain boundaries of a nanophase material as a function of grain diameter, with grain boundary thickness of 0.5 and 1.0 nm (i.e., 2 or 4 atomic planes). [R.W. Siegel, Annu. Rev. Mater. Sci. 21, 559 (1991)]

ATOM LOCATIONS
-NANOPHASE MATERIALS-



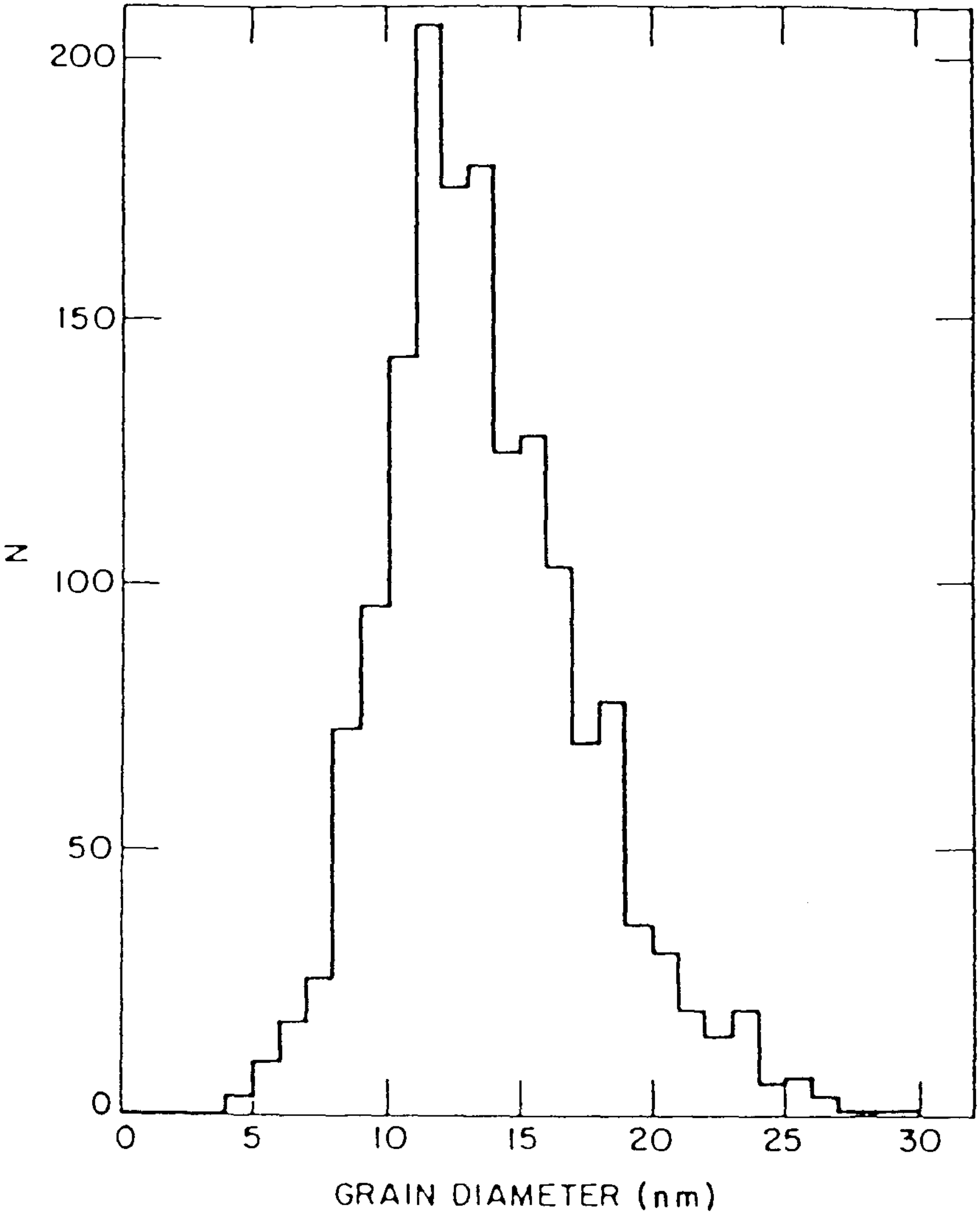
The effect of grain size on the calculated volume fractions for intercrystalline regions, grain boundaries, and triple junctions, assuming a grain boundary thickness of 1 nm. [C. Suryanarayana and F.H. Froes, Met. Trans. A23, 1071 (1992)]

VAPOR-CONDENSATION PROCESS



Schematic of a gas-condensation chamber for the synthesis of nanocrystalline materials. [R.W. Siegel, MRS Bulletin 15, 60 (1990)]

VAPOR-CONDENSED PARTICLE SIZE DISTRIBUTION



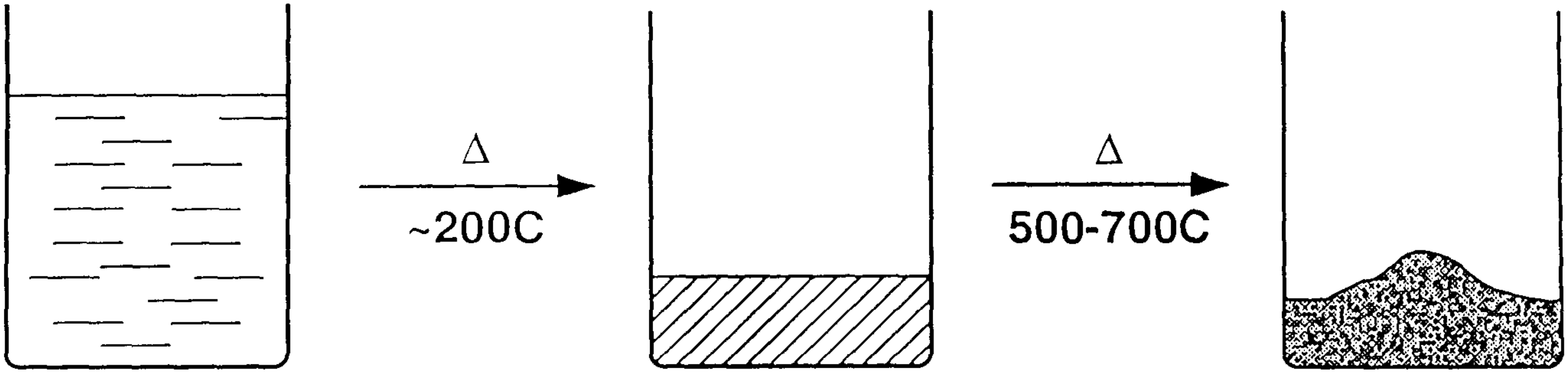
Grain size distribution for nanophase TiO_2 (rutile) sample compacted to 1.4 GPa at room temperature as determined via TEM. [R.W. Siegel, S. Ramasamy, H. Hahn, Z. Li, T. Lu, R. Gronsky, J. Mater. Res. 3, 1376 (1988)]

CHEMICAL ROUTES TO NANOPHASE AND NANOCOMPOSITE MATERIALS AT NIST

1. "Modern" Chemical Procedures
   - (a) Sol–gel techniques
   - (b) Controlled Pyrolysis of metal–organo complexes
   - (c) Reductive dehalogenation

2. "Classical" Chemical Procedures
   - (a) Coprecipitation
   - (b) Ion reduction in solution
   - (c) Use of oxidizing, reducing, nitriding, dehalogenating, etc., gases

METAL ION - ORGANIC COMPLEX ROUTE



Δ

~200C

Δ

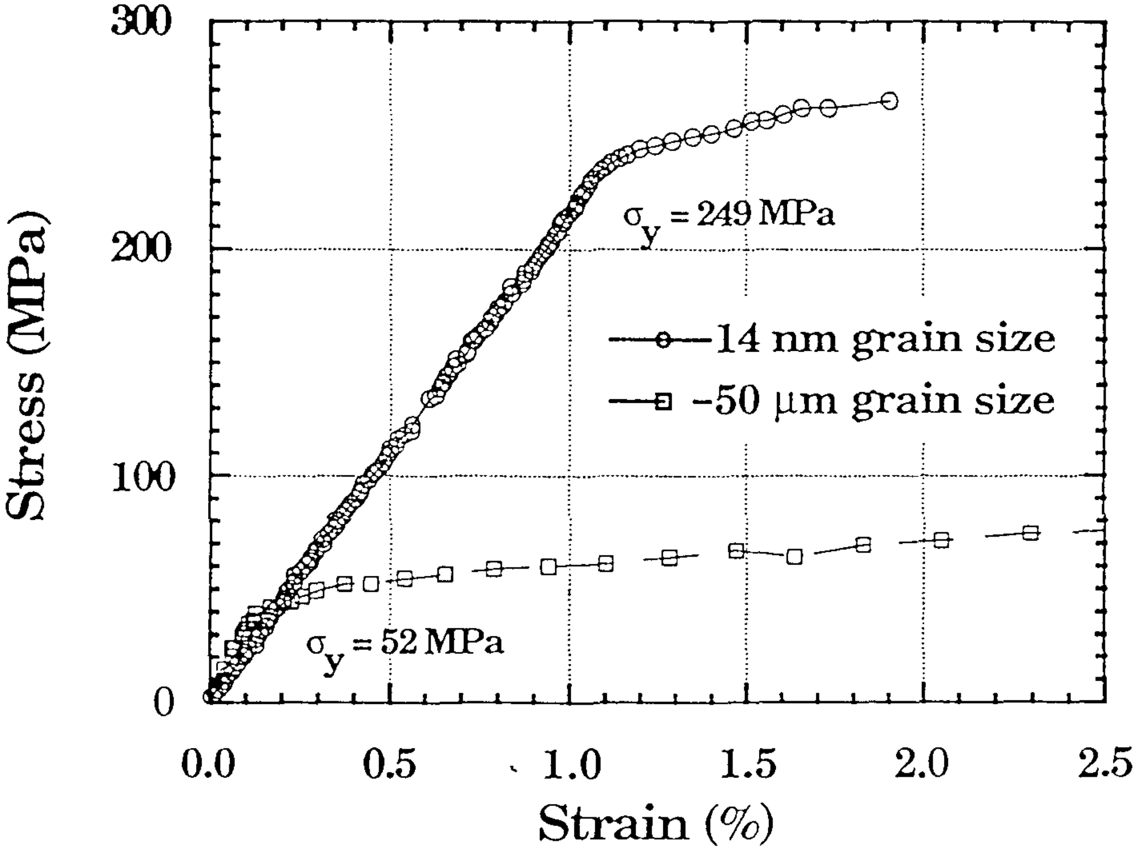
500-700C

Soluble Metal ion-
Organic Molecule
Complexes

Polymer

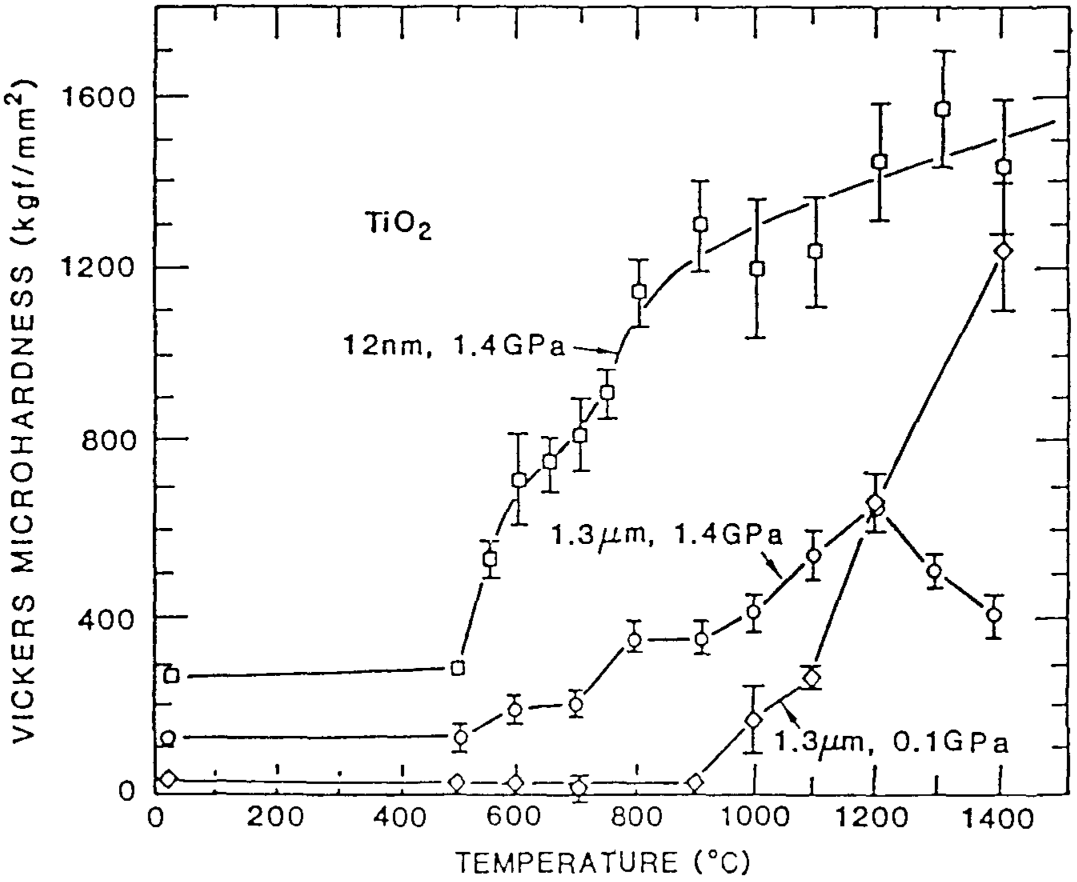
Multicomponent
Oxide Powder

INCREASED YIELD POINT



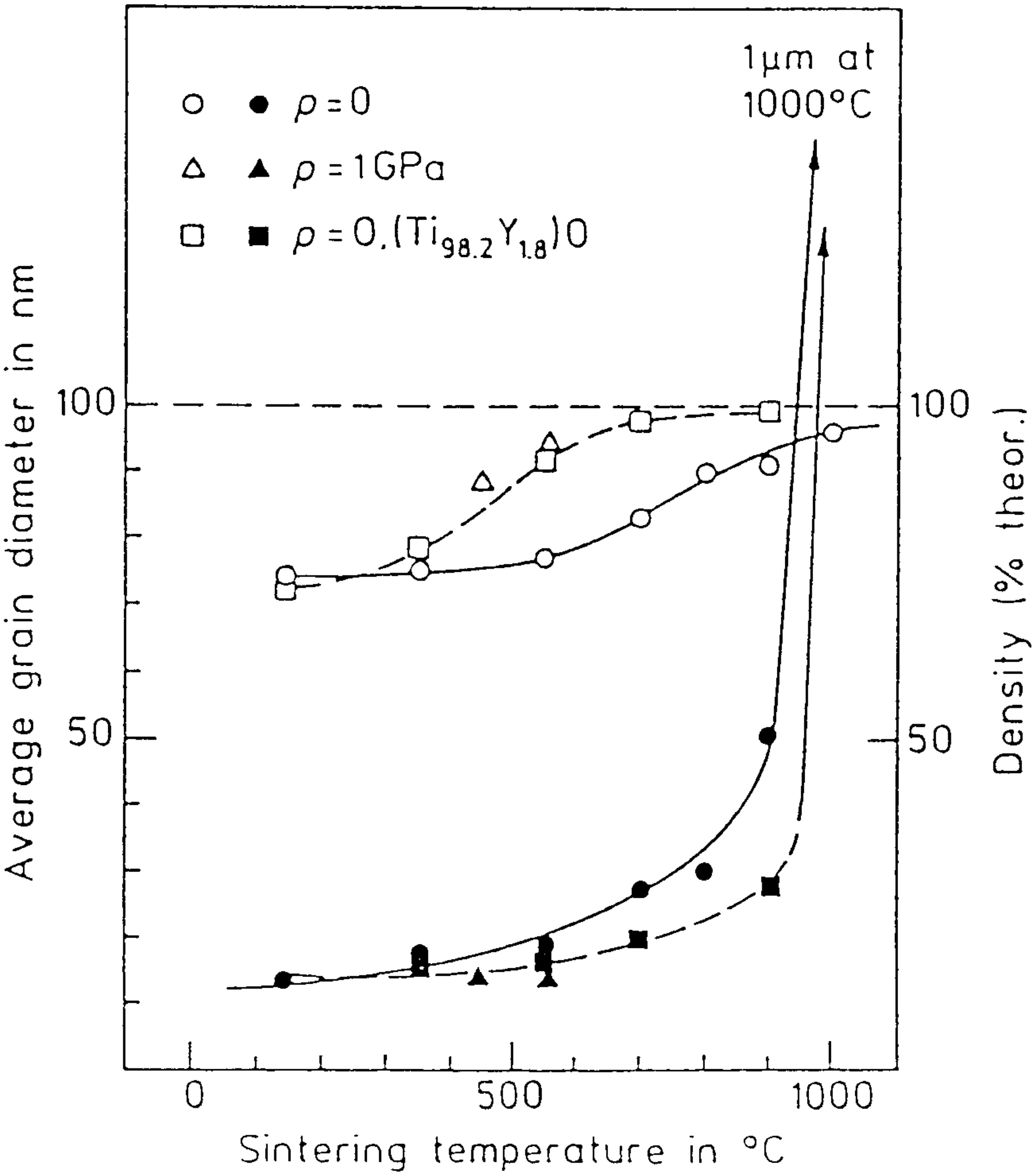
Stress-strain curves for nanocrystalline (14 nm) and coarse-grained (50 μm) Pd, measured at a strain rate of $2\text{x}10^{-5}$ s^{-1}. [G.W. Nieman, J.R. Weertman, & R.W. Siegel, Scripta Metall. Mater. 24, 145 (1990)]

LOW TEMPERATURE SINTERABILITY



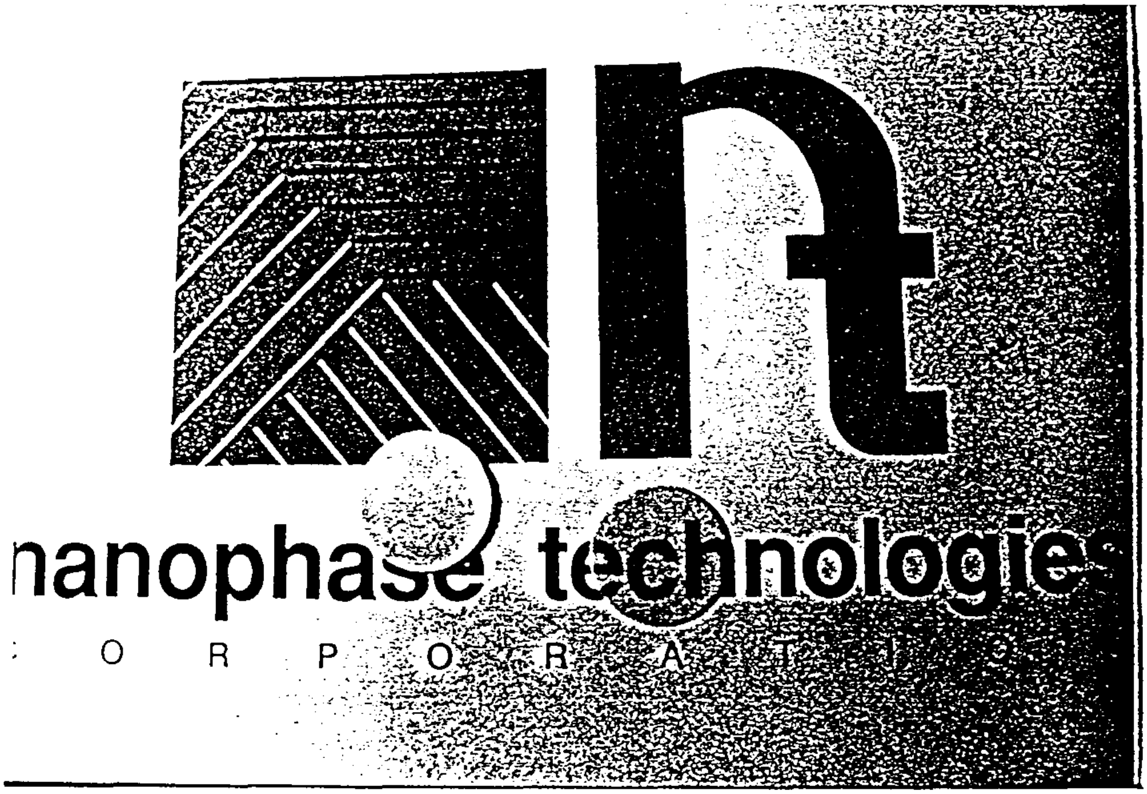
Vickers microhardness of TiO_2 (rutile) measured at 300 K as a function of one-half hour sintering at the indicated temperatures. Note that the 12 nm grain size rutile sinters at temperatures 400-600°C lower than coarse-grained (1.3 μm) material. [R.W. Siegel, S. Ramasamy, H. Hahn, Z. Li, T. Lu, & R. Gronsky, J. Mater. Res. 3, 1376 (1988)]

GRAIN SIZE STABILITY



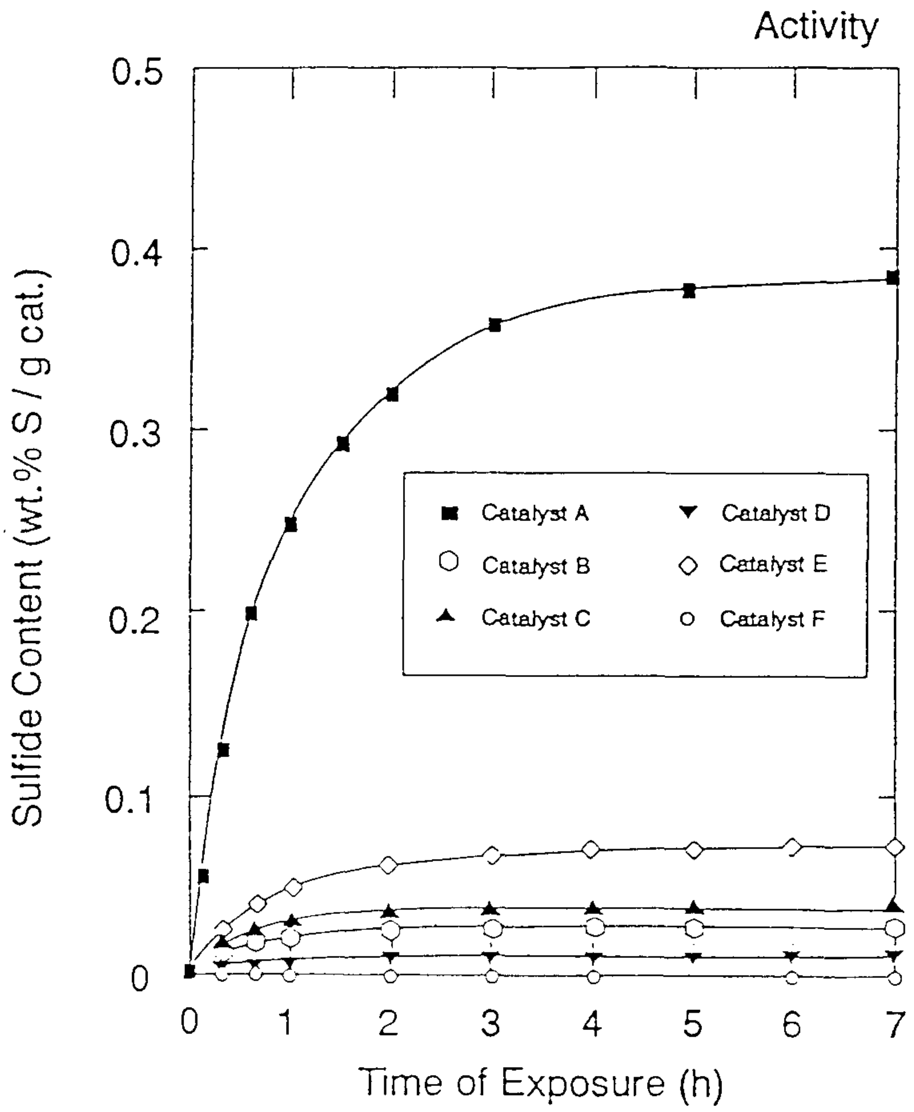
Density (open symbols) and grain size (filled symbols) of nanocrystalline TiO_2 compacted at the indicated pressures as a function of sintering temperature. [R.S. Averback, H. Hahn, H.J. Höfler, J.L. Logas, & T.C. Chen, Mater. Res. Soc. Symp. Proc. 158, 3 (1989)]

OPTICAL TRANSPARENCY



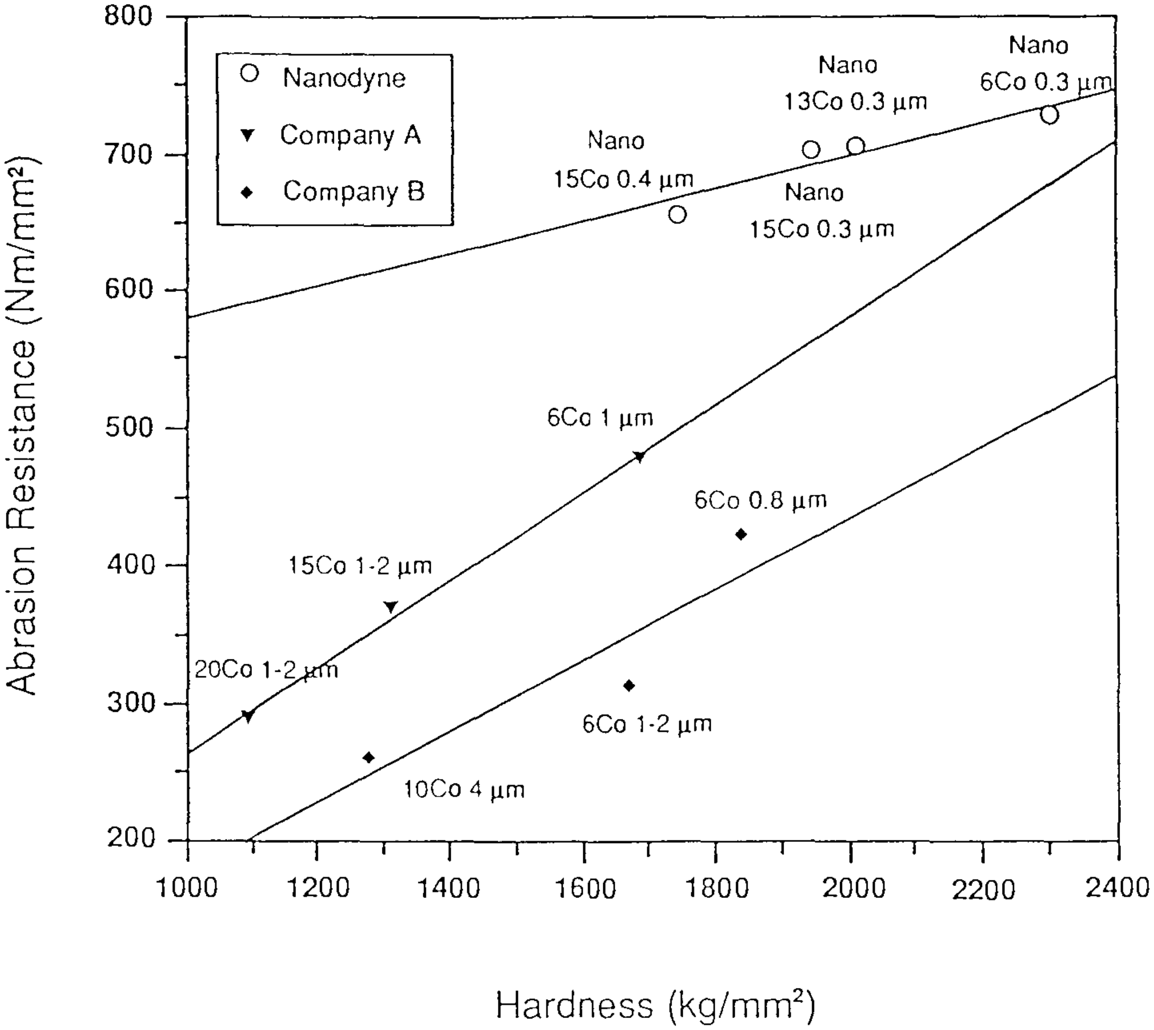
Disks of nanocrystalline Y_2O_3 showing that the optical transparency can be controlled by changing the pore size. (J.C. Parker, private comm. 1/94)

INCREASED CHEMICAL ACTIVITY



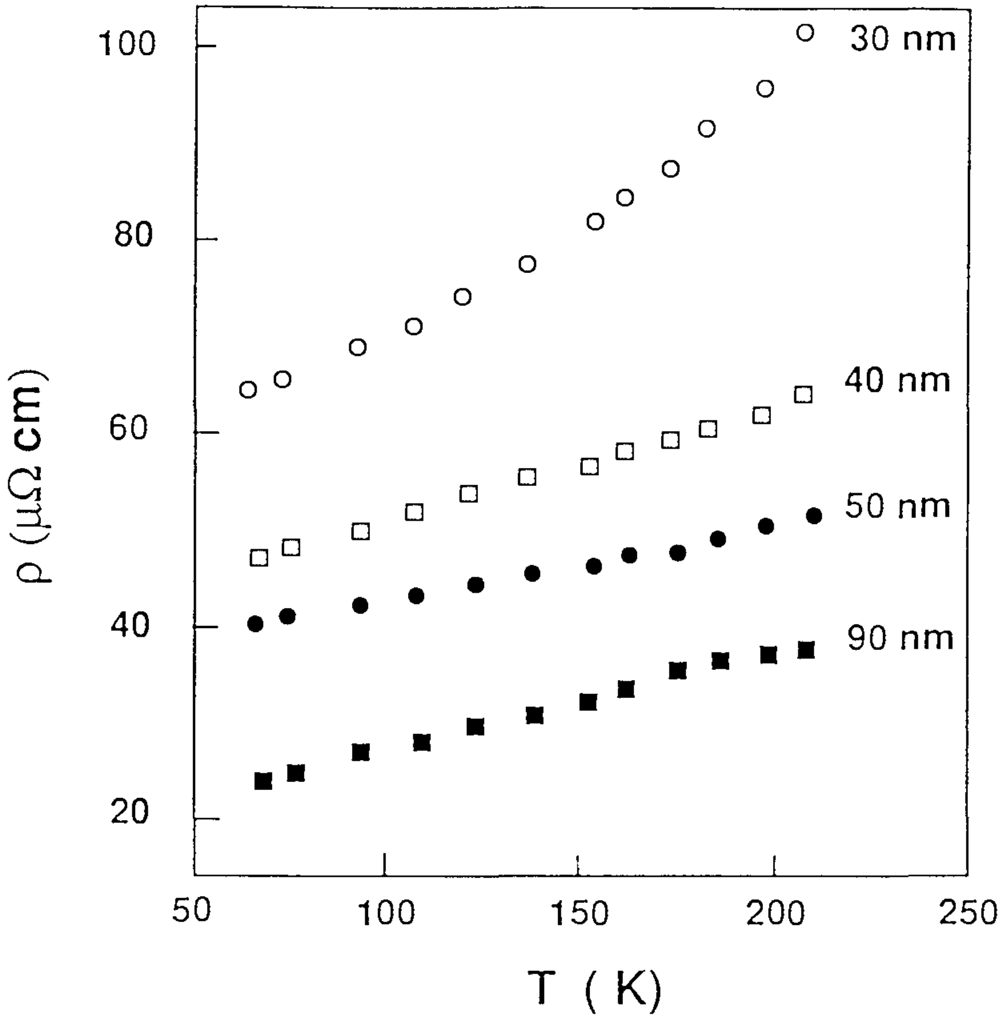
Activity of nanocrystalline TiO_2 for H_2S decomposition as a function of exposure time at 500°C compared with that from several commercial TiO_2 materials and a reference (A: 76 m^2/g nanocrystalline rutile; B: 61 m^2/g anatase; C: 2.4 m^2/g rutile; D: 30 m^2/g anatase; E: 20 m^2/g rutile; and F: reference alumina). [D.D. Beck and R.W. Siegel, J. Mater. Res. 7, 2840 (1992)]

INCREASED ABRASIVE WEAR RESISTANCE



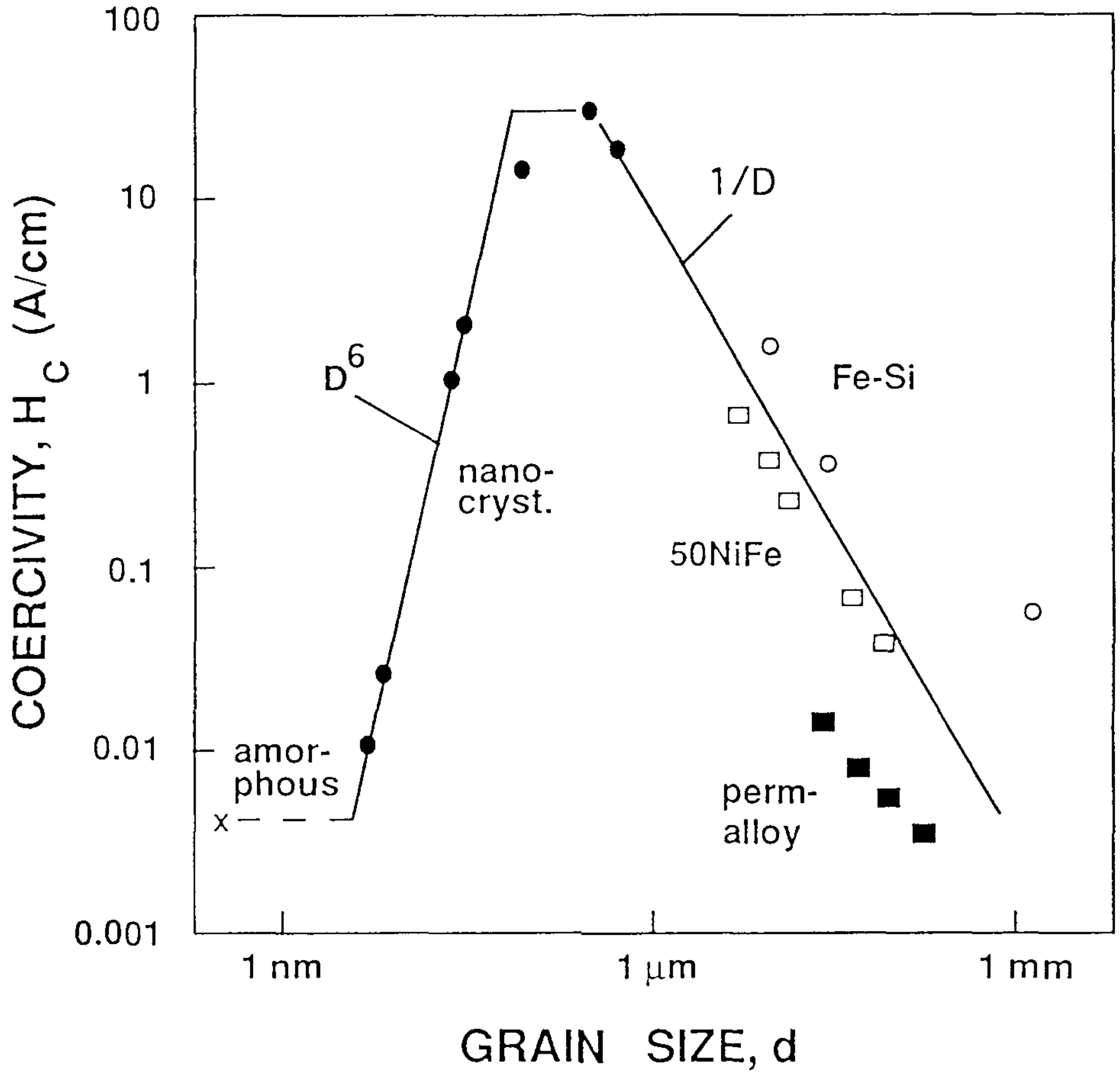
Abrasion resistance vs. hardness for conventional and nanocrystalline WC-Co materials. [L.E. McCandlish, private comm. 1994]

INCREASED ELECTRICAL RESISTIVITY



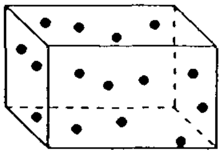
Variation of electrical resistivity with temperature for nanocrystalline Fe-Cu-Si-B alloys. [Y.Z. Wang, G.W. Qiao, X.D. Liu, B.Z. Ding, & Z.Q. Hu, Mater. Lett. 17, 152 (1993)]

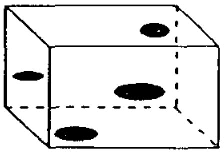
REDUCED MAGNETIC COERCIVITY

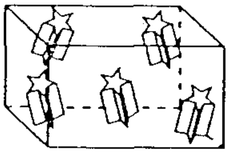


Magnetic coercivity vs. grain size for several soft ferromagnetic materials. [G. Herzer, IEEE Trans. MAG26, 1397 (1990)]

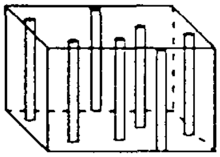
NANOCOMPOSITES

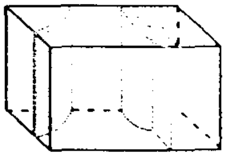

Spherical particle

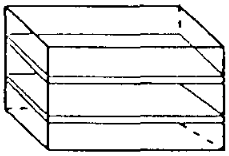

Disk-type particle


Rod-shaped particle

- Materials possessing a characteristic size scale on the order of ≈ **1-20 nm**
- For magnetic properties: critical lengths are grain size, magnetic species diameter (+ separation), and magnetic exchange length
- Are Composites of two or more magnetic states; highly composition dependent
- May be single or multiple phase
- Can be prepared from the vapor, from chemical solution, via precipitation, and by deformation
- Possess (1) UNIQUE properties, (2) NEW magnetic states, and (3) UNUSUAL property combinations
- Provide Opportunity for Electronic and Magnetic Engineering

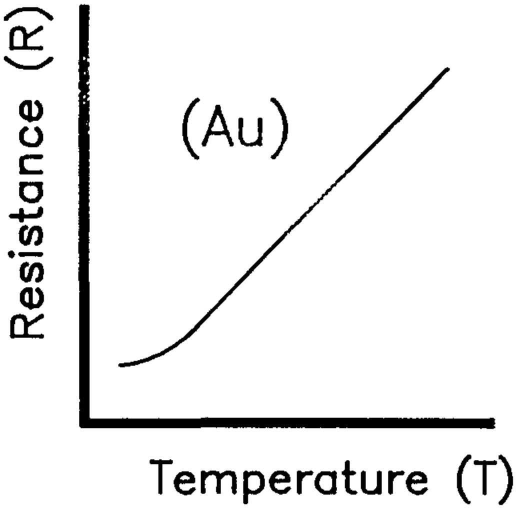

Fiber


Thin layer


Lamella

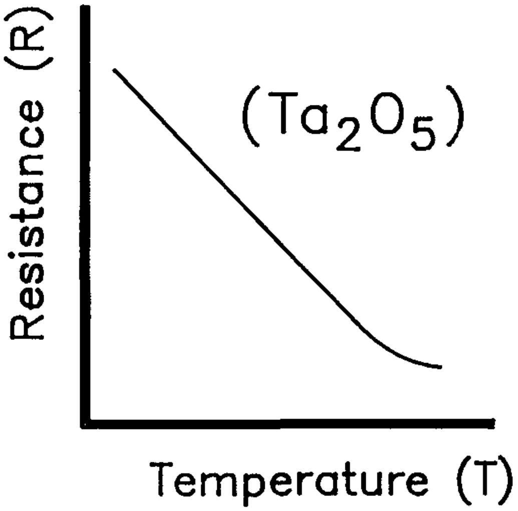
Zero Thermal Coefficient of Resistance

METAL



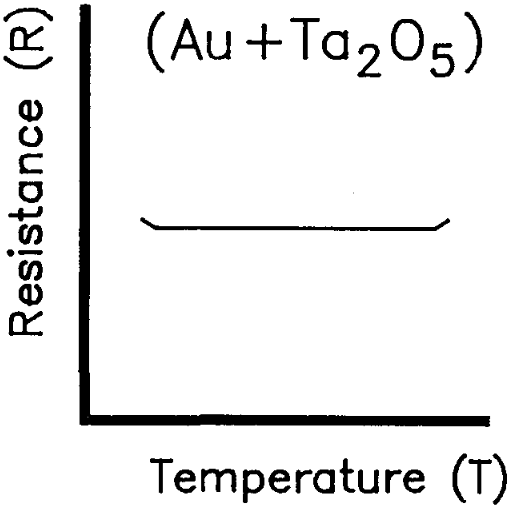
+

NON-METAL



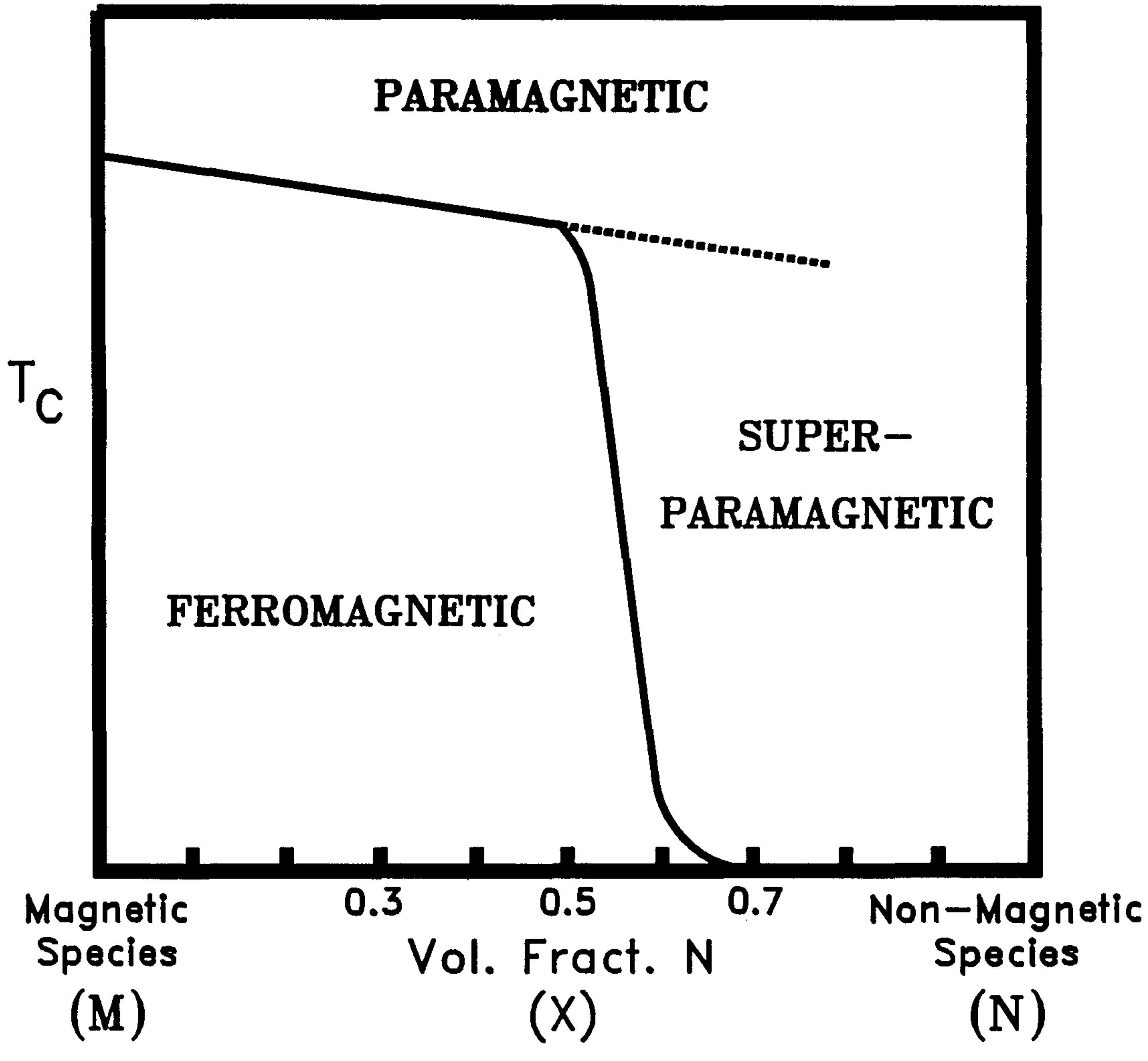
NANOCOMPOSITE

=

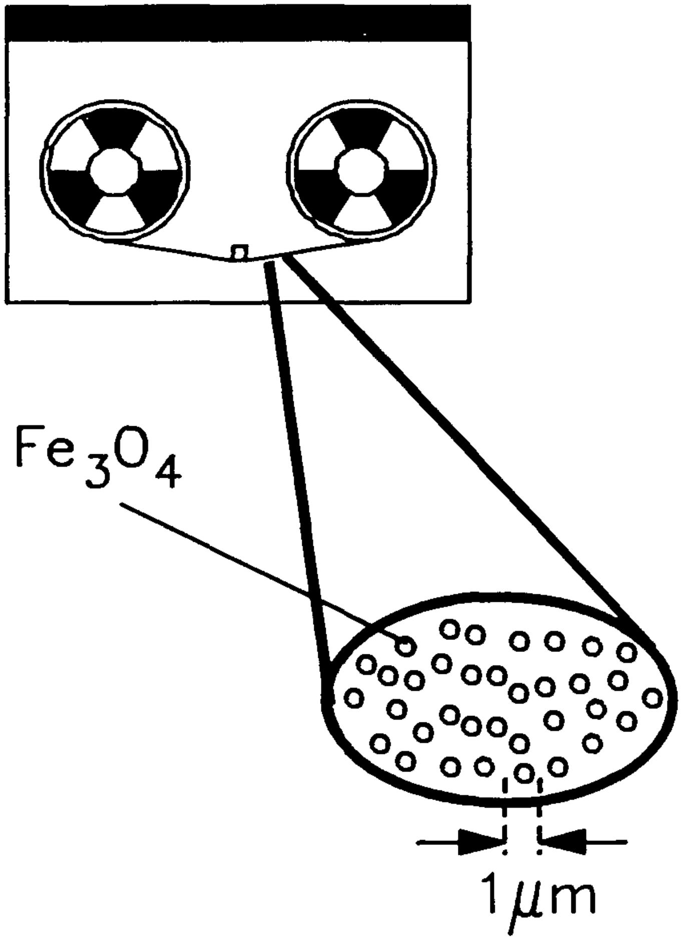


Resistors requiring NO WARMUP PERIOD

NANOCOMPOSITE
$(M_{1-x}N_x)$

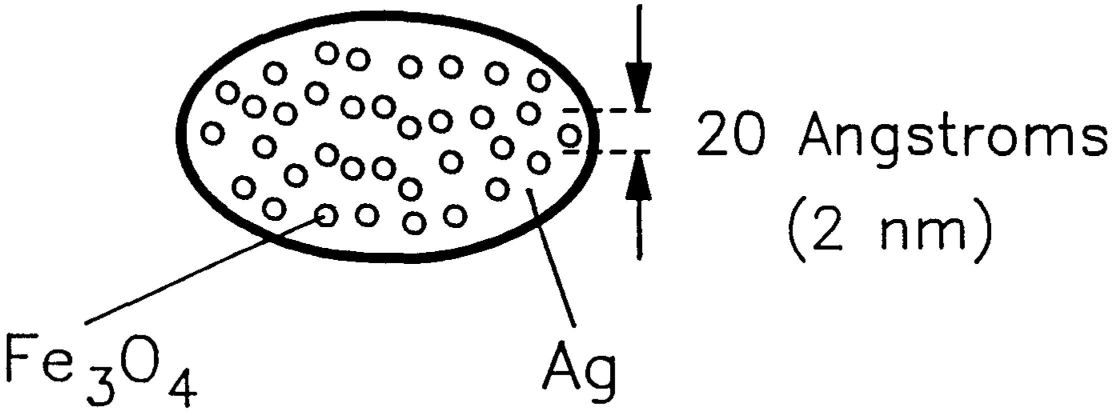


Conventional Magnetic Recording Media

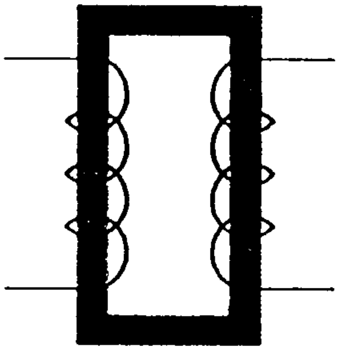


High Density Magnetic Recording Media

NANOCOMPOSITES



SOFT MAGNETIC MATERIALS



For Large Grains:

Magnetization (M) is determined largely by magnetocrystalline anisotropy (K)

For Small Diameters:

Increased importance of ferromagnetic exchange causes decrease in K

Nanocrystalline Fe–Cu–Nb–Si–B ("Finemet")

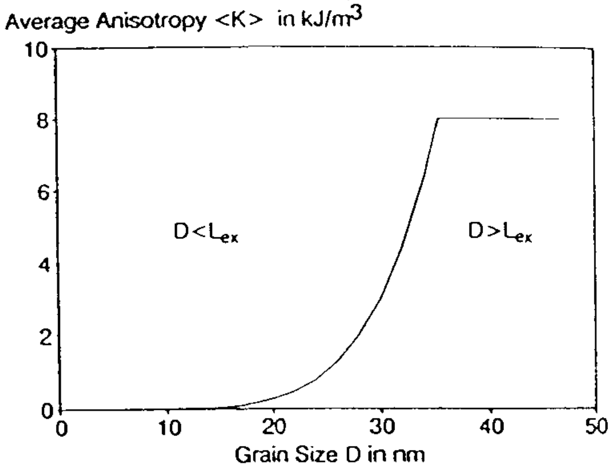
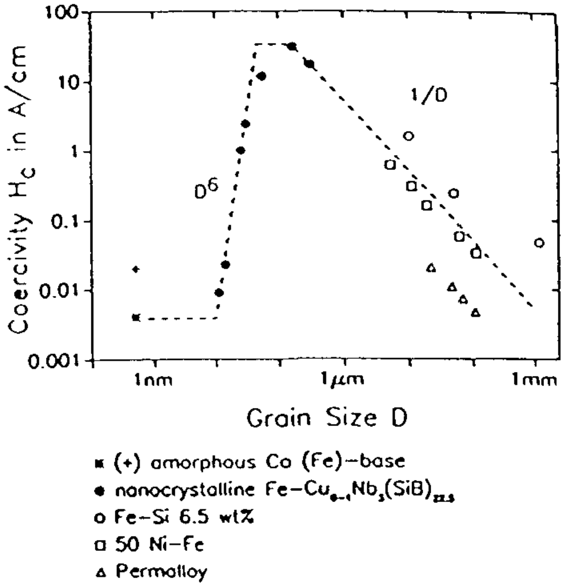
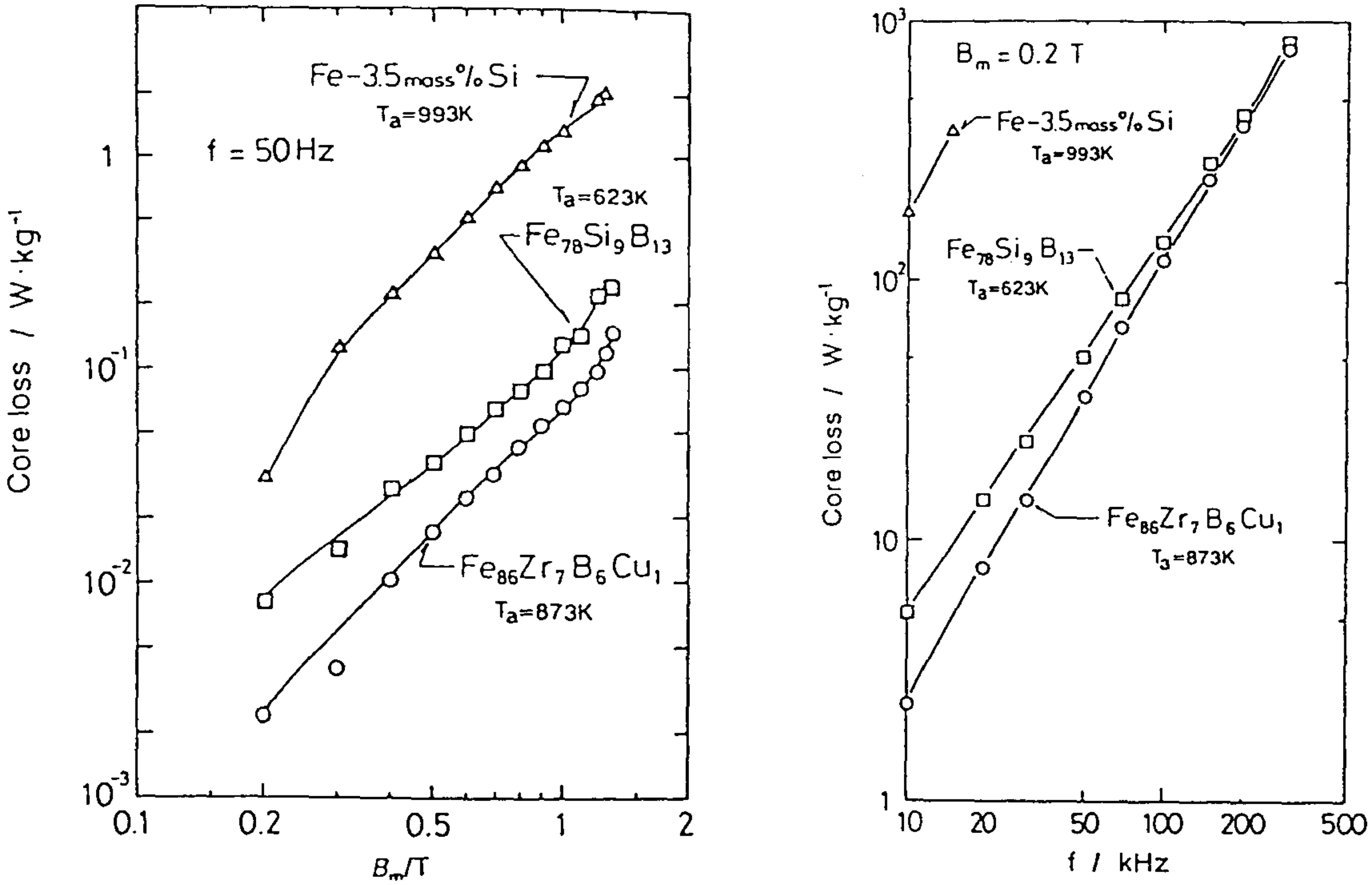


Fig. 7. Theoretical estimate of the average anisotropy ⟨K⟩ for randomly oriented α-Fe–20at.%Si as a function of grain size D.



G. Herzer, Mat'ls. Sci. & Eng. A133 (1991) p. 1.

REDUCED CORE LOSSES



Comparison of core losses of conventional coarse-grained Fe-3.5 wt.% Si, amorphous Fe-9 at.% Si-13 at.%B, and nanocrystalline Fe-7 at.%Zr-1 at.%Cu-6 at.%B alloys as a function of maximum induction field (B_m) and frequency (f). [A. Makino, K. Suzuki, A. Inoue, & T. Masumoto, Mater. Trans. JIM 32, 551 (1991)]

HARD MAGNETIC MATERIALS

"Magnequench" Magnets

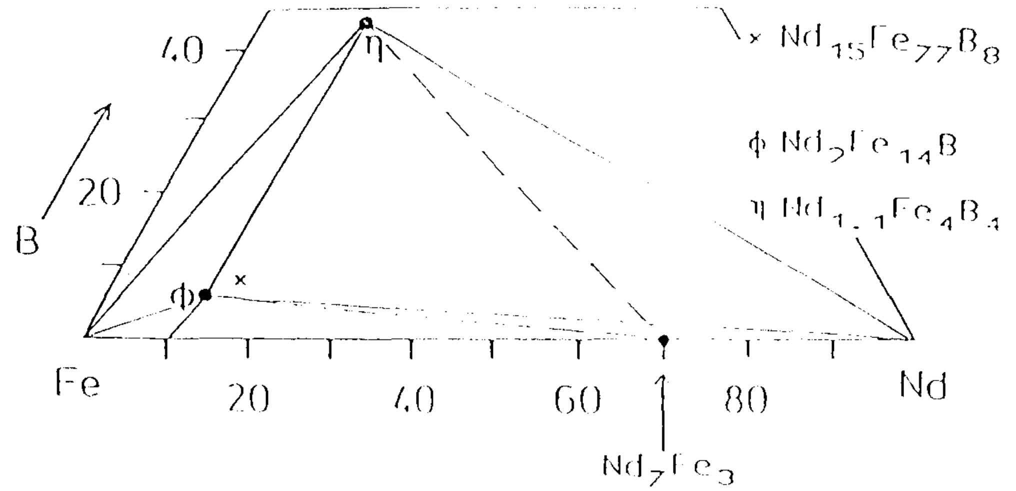


Figure 7. A comparison of the phase relations in the equilibrium phase diagram (solid line) and in the metastable phase diagram (dashed line).

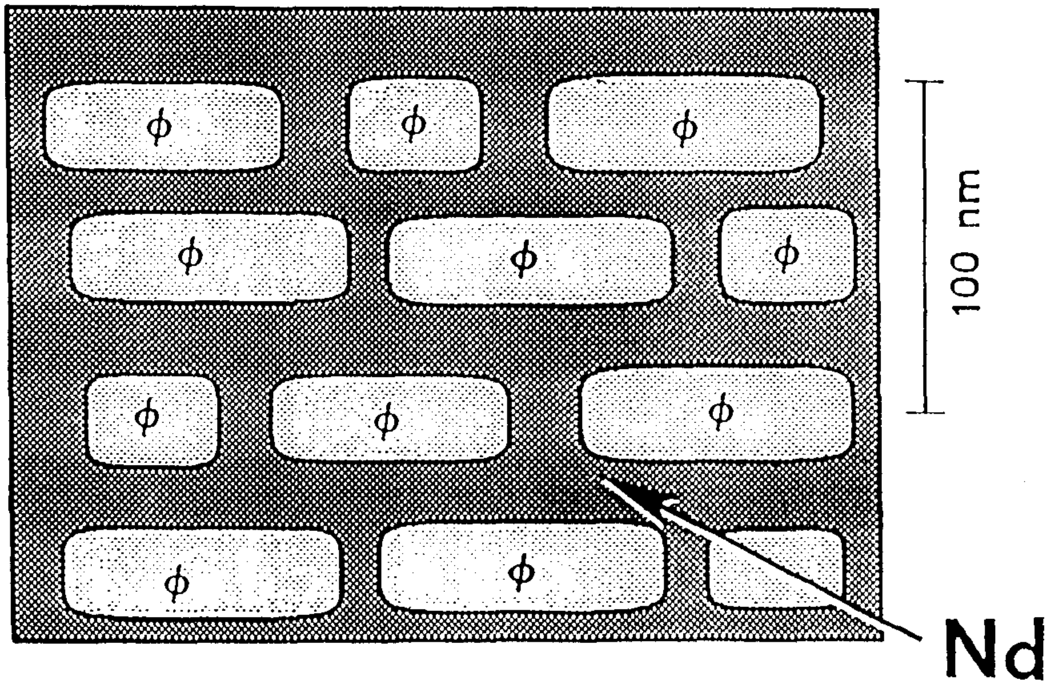
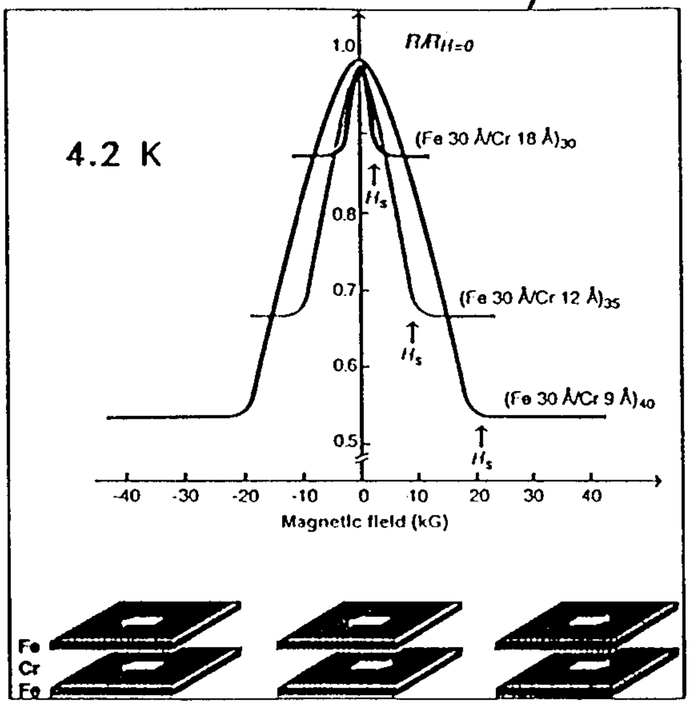


Figure 8. The ideal microstructure of a sintered magnet. The hard magnetic grains (ϕ) are decoupled by neodymium-rich nonmagnetic grain boundary phase.

A. Hutten, JOM (March, 1992) p. 11.

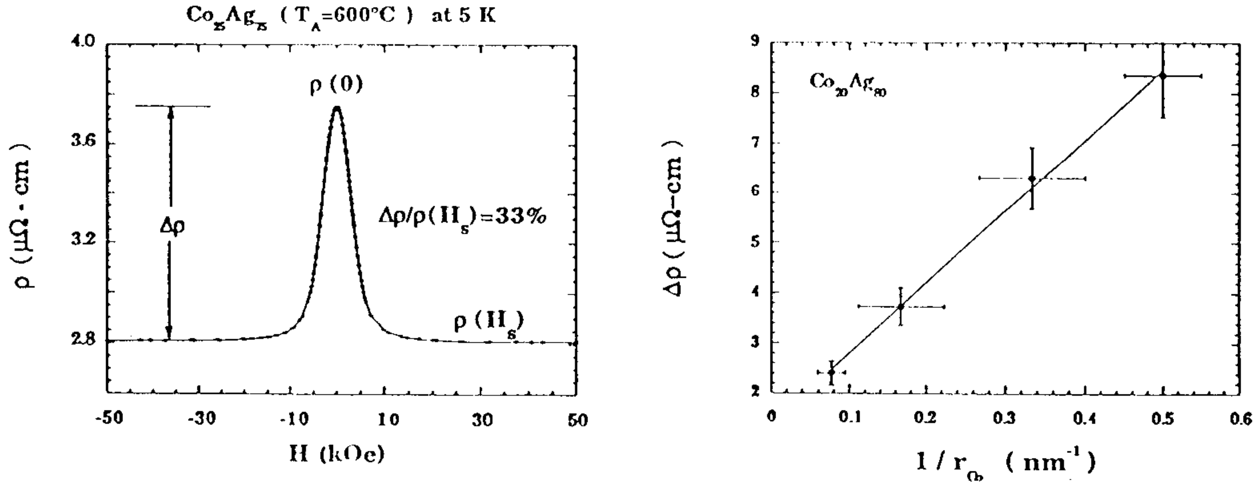
GIANT MAGNETORESISTANCE

Fe–Cr–Fe Multilayers



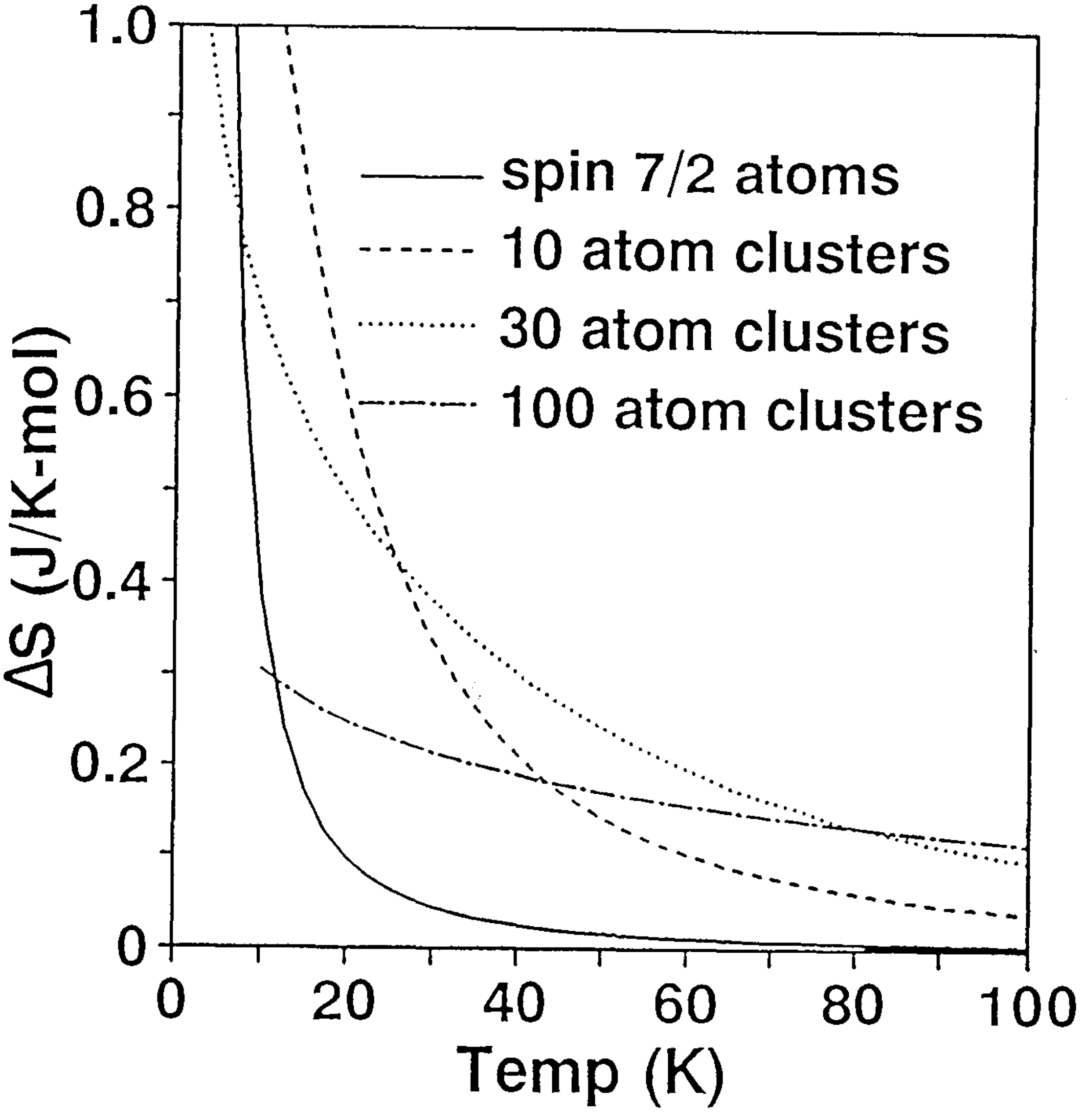
Baibich, et. al., Phys. Rev. Lett. 61 (1988) p. 2472.

Co–Ag Nanocomposites



Xiao, et. al., MRS Symp. Proc. 286 (1993) p. 197.

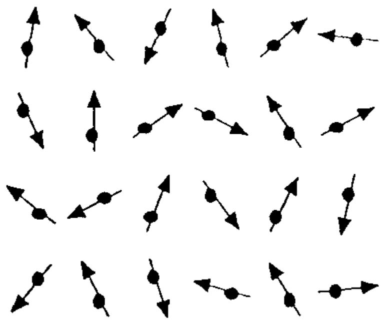
NEW MAGNETIC REFRIGERANTS



Calculated entropy change vs T for a magnetic field change of 1 tesla for a system of magnetic spins isolated and grouped into clusters as in magnetic nanocomposites. Note the enhancement when clustered. [R.D. McMichael, R.D. Shull, L.J. Swartzendruber, L.H. Bennett, & R.E. Watson, J. Mag. & Magn. Mater. 111, 29 (1992)]

MAGNETOCALORIC EFFECT

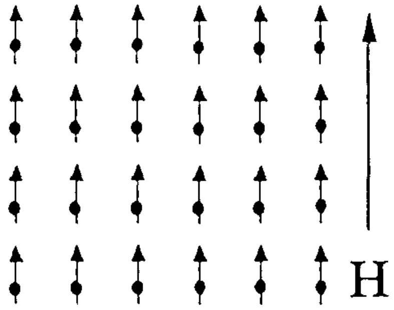
High Spin Entropy



Zero Field

Adiabatic application of a magnetic field (H) lowers the magnetic spin entropy and heats the sample. Removal of the field cools the sample.

Low Spin Entropy

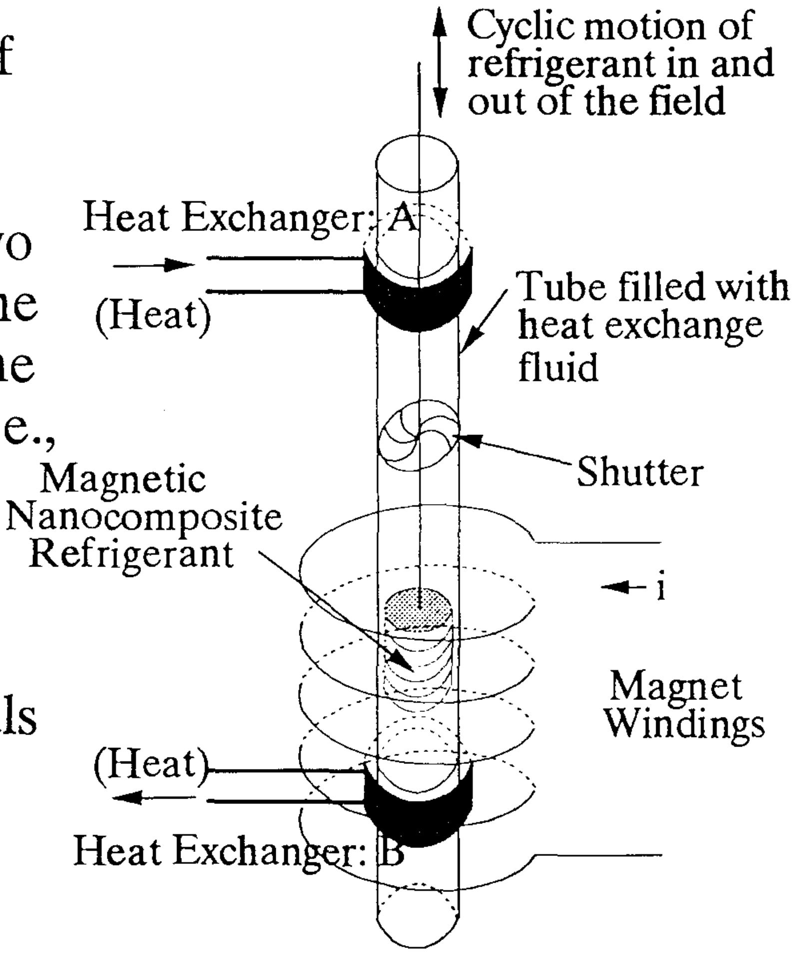


Applied Field

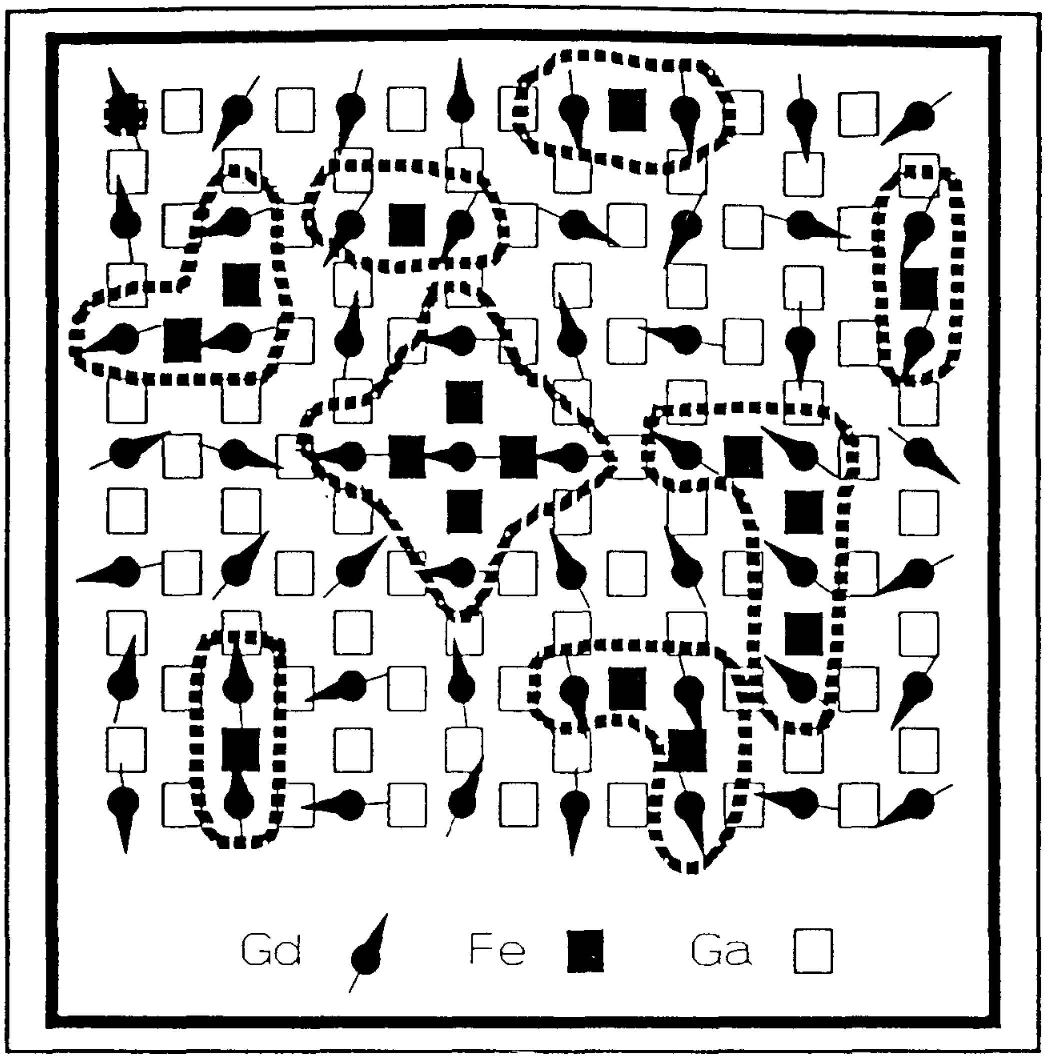
MAGNETIC REFRIGERATOR

Cyclic motion of a refrigerant in and out of a magnetic field while alternating the thermal connections between two heat reservoirs allows the transfer of heat from one reservoir to the other: i.e., a refrigerator.

General Motors Corp. is working with NIST to develop new materials for higher temperature cooling jobs, such as car air conditioners.

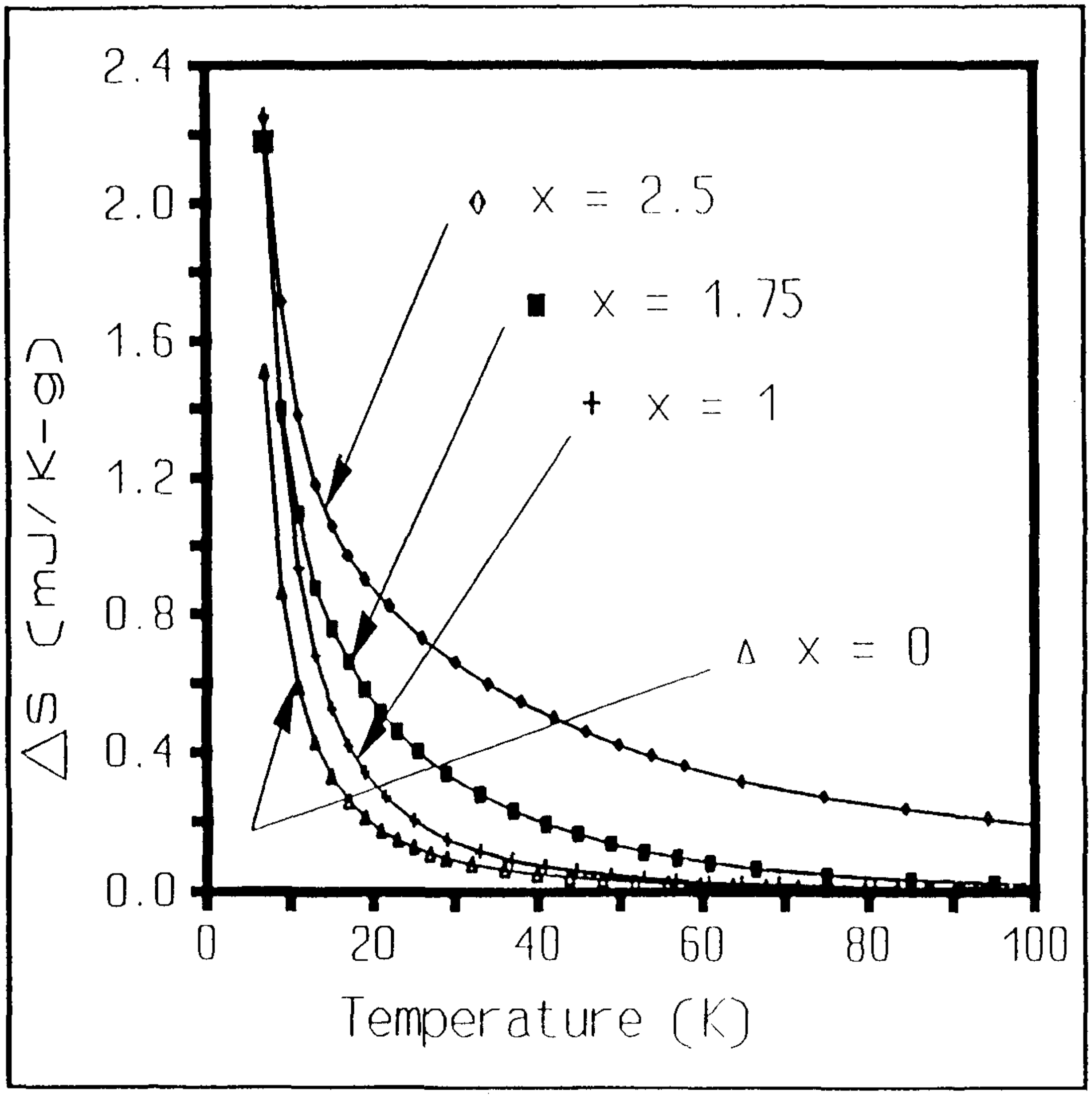


SINGLE-PHASE MAGNETIC NANOCOMPOSITE REFRIGERANT



Schematic of the $Gd_3Ga_{5-x}Fe_xO_{12}$ magnetic nanocomposite structure showing atomic positions, spin directions (via arrows) of the Gd atoms and the locations of magnetic clusters (dashed lines). [R.D. Shull, R.D. McMichael, J.J. Ritter, and L.H. Bennett, MRS Symp. Proc. 286, 449 (1993)]

MEASURED MAGNETOCALORIC EFFECT FOR $Gd_3Ga_{5-x}Fe_xO_{12}$ NANOCOMPOSITES



Measured entropy changes (for a field change of 1 tesla) vs T for paramagnetic GGG (x=0) and magnetic nanocomposites GGIG (x>0) showing enhanced magnetocaloric effects of the nanocomposites. [R.D. Shull, R.D. McMichael, J.J. Ritter, and L.H. Bennett, MRS Symp. Proc. 286, 449 (1993)]

DEVELOPMENT OF COURSE MODULES FOR MATERIALS EXPERIMENTS

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and

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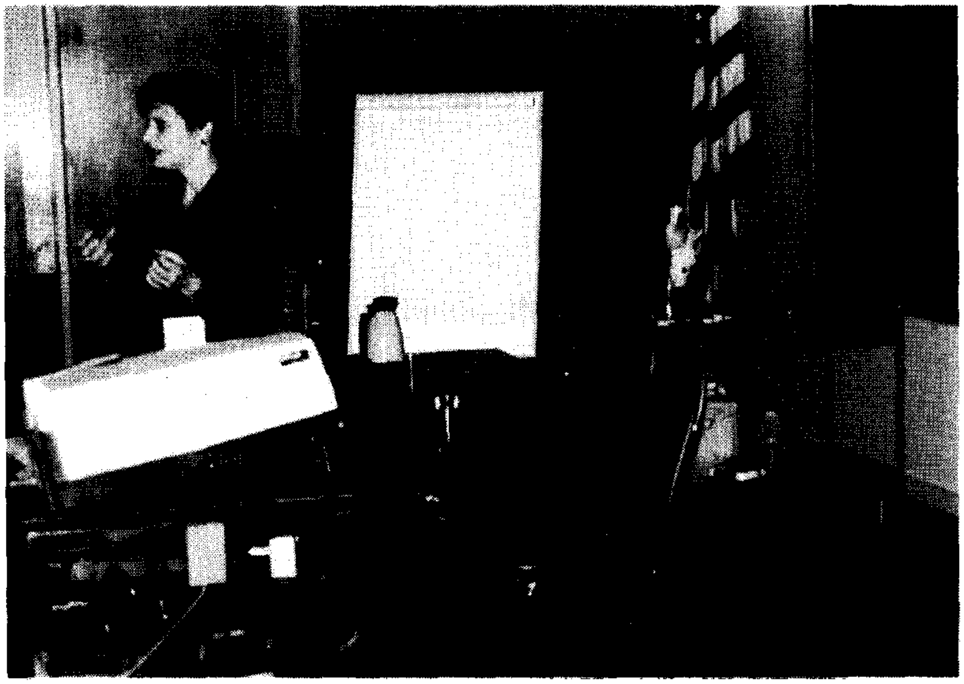

DEVELOPMENT OF COURSE MODULES FOR

MATERIALS EXPERIMENTS

K.P. Constant and K. Vedula
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Iowa State University
3053 Gilman Hall, Ames, IA 50011

Key Words: Computer Courseware

Prerequisite Knowledge: Basic math, chemistry and physics for entry-level materials science laboratories, elementary computer skills.

Objective: To enhance learning in materials science laboratories.

Equipment: This courseware is presently Macintosh compatible, although a PC version will be available in the near future. The minimum system configuration is system 7.0 or higher, 4 MB of RAM, and prefers 256 colors.

Introduction:

Recent U.S. government-sponsored studies of the decline of international competitiveness have reported that one of the most severe problems is the lack of education of future engineers in synthesis and processing of advanced engineered materials. In response, the National Science Foundation has made awards 6 universities to develop materials processing course materials for improving processing education. Iowa State University is among those selected.

Approach:

Development philosophy

A common problem with introducing new course material to an existing curriculum is that most programs are considered to be "over-committed" in terms of course topics and time and have very little flexibility. Few departments can afford the curriculum space to introduce an entirely new course in their program, and therefore must depend on introducing new or expanded material in the framework of the already existing courses. Since there is an outstanding variety of programs which include materials science and processing, flexibility is the key. In order to ensure that the materials developed at ISU can benefit the maximum number of students in Materials Science and Engineering, a new approach was developed.

A system involving flexible teaching modules has been designed to respond to the varying needs of existing departments, including that at Iowa State University. Each module is a 4-6 week set of teaching lectures and laboratories which can be used individually or together. This approach addresses the needs not only of the education of students majoring in the field of materials science and engineering, but also in allied disciplines either in the form of service courses taught to non-majors, or materials-related courses taught by other departments.

Computer-based course development was selected as a mode of delivery largely because of its flexibility. Computer based course material (courseware) can be designed such that the modules emphasizing specific processing techniques share common links to fundamental concepts so that each module can be as complete and concise as possible without repeating common material. Courseware also allows great flexibility in the order of presentation of materials, a significant departure from the traditional linear textbook approach. Also, the user can choose the level of the presentation, from basic to advanced. Delivery of courseware over a national network also provides maximum availability to students and educators. Additionally, courseware provides functionality not easily achieved in "hard copy" form. Specifically, color photographs and images, animations, videos, and sounds are easily accommodated. Finally, courseware can be designed to require interactive learning -- the student is asked to answer questions or provide some sort of input in order to proceed. It has been suggested that active learning is more effective for a majority of students than classical passive learning styles frequently associated with lectures or reading a text. Since this interactive component is particularly well-suited to laboratory exercises, the scope of lab exercises has been expanded to include 9 experiments outside the area of materials processing for use in an introductory materials science class.

Software description

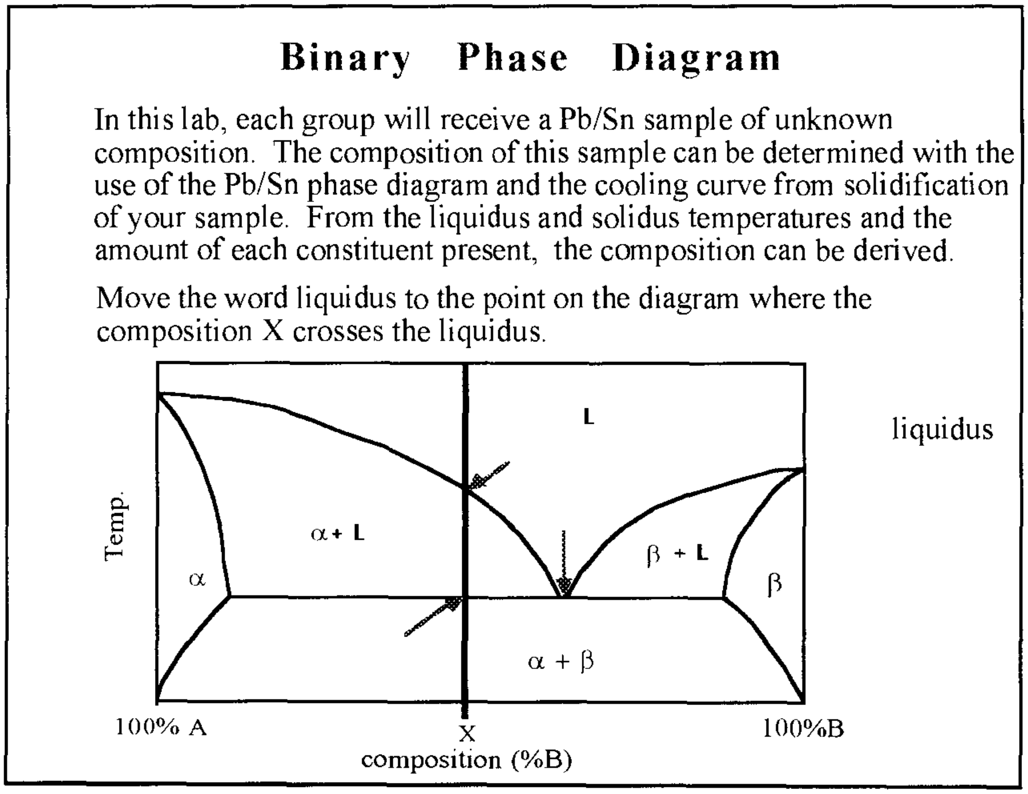
Authorware Professional™ 2.0.0 courseware development software was selected as a vehicle for delivery of these modules for a number of reasons. This software is available for both the Apple Macintosh and IBM-PC compatible platforms, and can be translated from the Macintosh to the PC, utilizing a large fraction of educational computing equipment already in place. This software also allows the "packaging" of programs so that the end product is self-contained. The user does not need to own the software in order to use the product. This software also has a full range of multi-media capabilities.

Navigating through the courseware is through pull-down menus and click-touch areas similar to those familiar to most students who have worked either in the Macintosh or Windows-environments. Within each section, push-button forward and backward arrows allow the students review material already presented. There is an optional "help session" available either at the beginning of a session, or at any time during the session which fully describes the use of the program. In the processing courseware, there are module-specific problems and experiments. The laboratory exercises make use of a variety of interactions including "assembly" interactions where a student is asked to drag and drop items with the mouse (either labels or pictures) to an appropriate place on the screen (sometimes in a specific order). This is extremely useful for "walking through" a lab experiment and equipment use before going in to lab. The experiments have an introduction, a background, a procedure and an analysis section, as well as information on safety and handling of the equipment and materials used.

The effectiveness of this approach is being tested this semester in an introductory materials science laboratory at Iowa State University. Students are required to complete a tutorial on the computer before attending the laboratory. One of the major advantages over other types of pre-lab exercises (in written form, or on video tape) is the ability of the computer to provide feedback both to the student and to the professor. Built into the software is a record tracking scheme so that a professor can access a file that records which students have accessed the file and what responses were given for various interactions. This provides a unique opportunity for the instructors to learn which parts of the laboratory are most confusing to students so they can provide additional information before and during the laboratory

Sample experiment:

Twelve experiments have been developed covering various concepts in structure of materails, phase equilibria, defects and electrical, thermal, and mechanical properties. One such experiment introduces phase diagrams through the use of the Pb/Sn system. The experiment involves identifying the composition of a Pb/Sn alloy by measuring the cooling curve and consulting the phase diagram. A number of concepts are employed, including binary phase diagrams. the lever rule and the phase rule. In the computer lab-tutorial the student completes exercises which describe phase diagrams including the level rule, the amount of phases and their compositions, and the phase rule. These exercises involve the student by requiring moving labels to appropriate areas, or by pointing to correct answers or by typing responses The student is then quizzed on various aspects of binary phase diagrams. Figure 1 shown one screen which requires the student label various parts of the phase diagram as they flash on the screen.



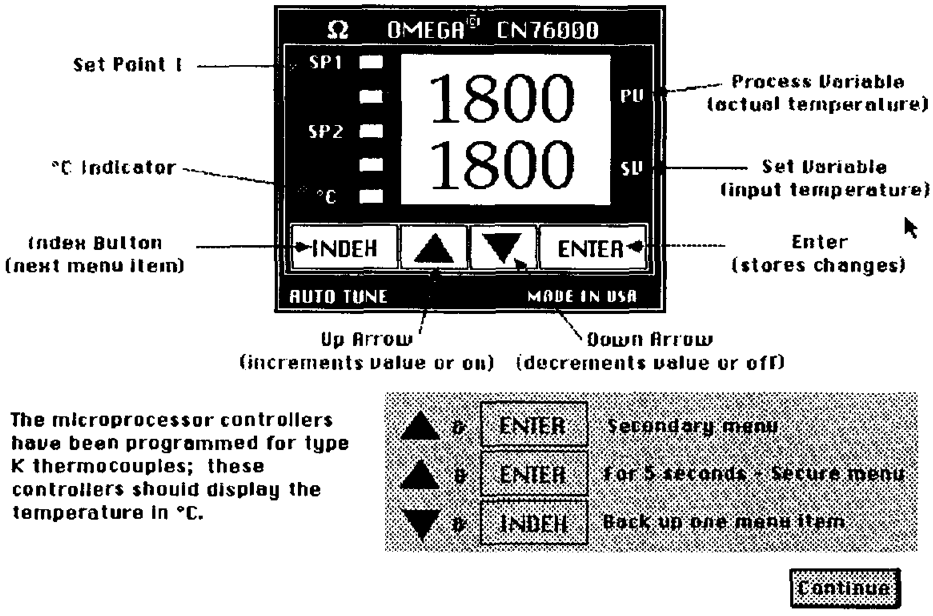
Binary Phase Diagram

In this lab, each group will receive a Pb/Sn sample of unknown composition. The composition of this sample can be determined with the use of the Pb/Sn phase diagram and the cooling curve from solidification of your sample. From the liquidus and solidus temperatures and the amount of each constituent present, the composition can be derived.

Move the word liquidus to the point on the diagram where the composition X crosses the liquidus.

Figure 1 A screen which shows the drag and drop interaction. Students are asked to name various parts of the phase diagram.

After the introduction, the experimental procedure is reviewed. In this section, students are required to assemble the apparatus for measuring the temperature of the alloy on cooling. Students may choose to review the use of the microprocessor based controllers used in this laboratory, if necessary. (Figure 2). Finally, typical results are shown and the analysis is discussed. (Figure 3)



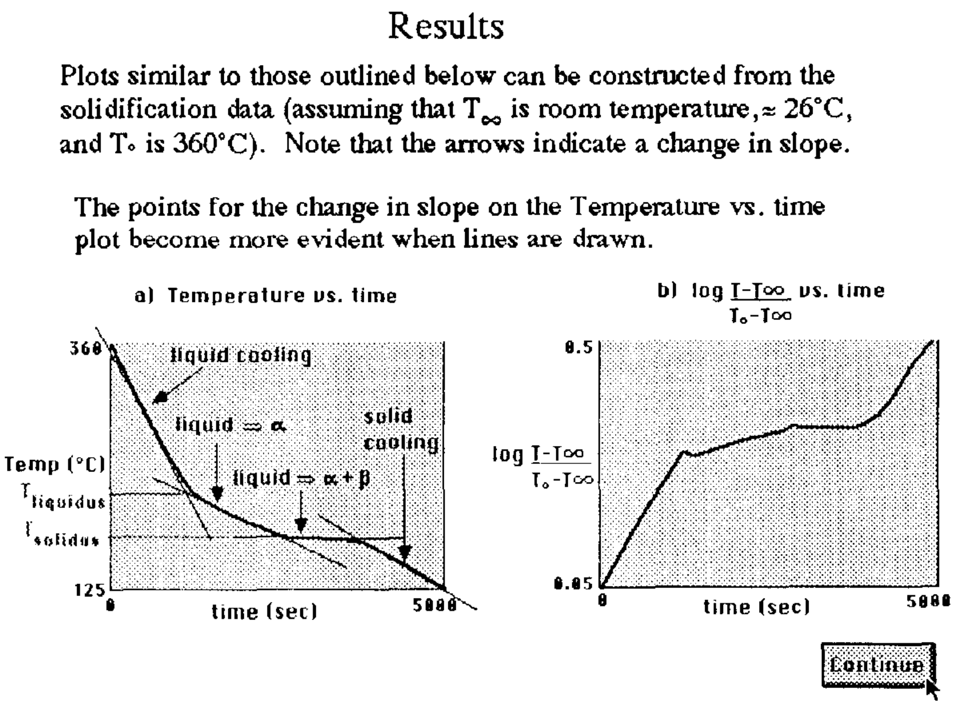
The microprocessor controllers have been programmed for type K thermocouples; these controllers should display the temperature in °C.

▲ & ENTER Secondary menu

▲ & ENTER for 5 seconds - Secure menu

▼ & INDEX Back up one menu item

Figure 2 A screen from the tutorial on using furnace controllers.



Results

Plots similar to those outlined below can be constructed from the solidification data (assuming that T_∞ is room temperature, ≈ 26°C, and T_o is 360°C). Note that the arrows indicate a change in slope.

The points for the change in slope on the Temperature vs. time plot become more evident when lines are drawn.

Figure 3 The Results screen shows the student typical results

Constraints and Challenges:

Dual Modes of Use

These experiment modules necessarily rely on the availability of specific equipment. Although we have made an attempt to include general equipment which is probably available in most materials science departments, it is quite likely that some of the experiments cannot be performed in some departments. We include sample results and reports for experiments which will provide some learning experience (though not hands on), for students in equipment-limited situations. In the case where equipment is available, the professor may choose to limit the students' access to the results and interpretation section to allow the student to perform these tasks alone.

Module Size

Multimedia computing can provide a rich experience of sight and sound, but can also require sophisticated hardware and large quantities of computer memory. We are attempting to limit file size and use file-compression schemes which would allow even modestly configured computers to use most, if not all of the capabilities of the modules.

Copyright issues

Although these modules will not be sold, there are copyright issues which must be considered especially for use of graphics. Since the laws relating electronic duplication and manipulation of images are not well defined, we have been keeping careful records of the sources of graphics used in these modules. We will seek guidance on these legal aspects, and seek permission when necessary.

Assessment of Effectiveness

Although it is difficult to assess the effectiveness of these lab modules in enhancing the laboratory learning experience, we hope to learn from student responses to questionnaires. We also plan to test this material at other universities and solicit suggestions for improvement.

Future Work:

Work on experimental modules will continue both improving existing modules and expanding the scope of experiments to include a broader range of advanced materials. A module involving laboratory safety is also planned.

This work is funded by the National Science Foundation and the Engineering Computing Support Services at Iowa State University

TESTING SAND QUALITY IN THE FOUNDRY (A BASIC UNIVERSITY-INDUSTRY PARTNERSHIP)

Donald H. Martin

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TESTING SAND QUALITY IN THE FOUNDRY (A BASIC UNIVERSITY - INDUSTRY PARTNERSHIP)

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Key Words: Sand, clay, bonding material, moisture content, grain size, permeability moldhardness, green compression, friability, sand temperature, compactibility.

Prerequisite Knowledge: No prerequisite knowledge required for this experiment.

Objective: To observe and understand the key elements in sand testing (specifically testing for moisture content) in a modern flaskless foundry operation. A look at sand, clay, and sand additive receiving control to reclaiming and controlling process sand.

Equipment: Equipment essential for making moisture tests with the No. 276 Moisture Teller is as follows:

1. The Moisture Teller
2. Lecture Type Balance, No. 250 or No. 240-A Speed Balance
3. Sample pans with wire cloth bottoms. (Two tared pans of matched weight are supplied with the No. 276 Moisture Teller.)
4. Counterweight (equal to the weight of the pans) and a 50 gram weight or a set of weights No. 260, for special work.
5. Pan Lifter for handling pans while hot and soft brush for cleaning wire cloth pan bottoms (supplied with all Moisture Tellers).

Equipment can be purchased from:

George Fischer Foundry Systems, Inc.
407 Hadley Street
P.O. Box 40
Holly, Michigan 48442

Introduction:

One of the most important elements in proper foundry sand conditioning is percentage of moisture content. Too much moisture and I've got "mud" and gas holes in the cast part; too little moisture and the sand will not hold its shape and crumble and you lose dimensional control of the part. There are many deposits of various grades of molding sands found throughout the world. The most common sands are composed of silicas, which do not melt or fuse together when they contact the molten metal in the casting process.

We talk about "green sand molding", but we should explain that the sand is not green in color, but green in process. Green sand molds are widely used in today's foundry practices. They are simply made of the sand, clay, water, and other materials and are used without further conditioning. Green means that the molded sand mixture is allowed to remain moist throughout the manufacturing process.

Silica sands are the most common types of sand used in the foundry operation today. Two general types are used: naturally bonded and synthetic. Both are found to exist naturally. Naturally bonded sands (bank sands) can contain up to 10-20% clay-based contaminants and must be selectively used depending on material being cast.

Synthetic sands (lake sands) are made up of basic sand grains. Most foundries processing metals at high temperatures use synthetic sands; the sand composition can be more closely regulated. In malleable and grey iron foundry operations a combination of naturally bonded and synthetic sands are used to provide quality casting.

Due to the shape of the grains and to some extent the amount of washing, wet-reclaimed sand usually appears grey. This is because of the retained carbon deposited on the sand grains.

The silica sand on the beach or bank sand will not hold its shape unless a bonding agent, usually clay of some type, is used to hold it together. We then add water (from 3 to 9%) depending on the material being cast and arrive at the required specifications for sand conditions required to consistently produce quality casting.

The size of pores (holes) in the face of molds varies depending on the sand size and shape and are determined by the mixture of various mesh sand sizes.

Clay is the most commonly used bonding material for molding sand. When clay is mixed with the correct amount (percent) of water, it is made plastic and adhesive and holds the sand grains together. When water is added to a mixture of sand and clay, all the water is absorbed by the clay. The shear strength of high-water clays is always low. As the amount of water decreases, the strength rises, slowly at first, but further decreases in water content cause a sharp rise in strength up to ideal sand specification (mixture).

Moisture determination of warm or hot sand needs special attention to enable the lab technician to obtain a moisture percentage that is meaningful. First, it must be realized that warm or hot sand is tempered with additional moisture to compensate for loss of moisture due to evaporation. The moisture percentage required at the sand mixer increases with an increase of sand temperature so that when the sand arrives at the molding station, the sand is not too dry, but contains an actual moisture percentage, which imparts a good moldability. Therefore, in sand control we are interested in two different moisture tests. One is the Compensated Moisture Test at the mixer, and the other is the Actual Moisture Test at the molding station.

In controlling the moisture at the sand mixer, we are interested in the compensated moisture. The compensated moisture test gives a moisture percentage that anticipates the moisture loss in transportation and storage. The compensated moisture percentage at the sand mixer is a constant. Whereas, if an actual moisture test was used showing all the moisture present, one would have a variable moisture percentage due to temperature fluctuations calling for different total moisture. Therefore, actual moisture percentage at the sand mixer would complicate sand control.

Most foundries are now using compensated moisture test readings, but are not fully aware of the meaning, nor are they conducting the test carefully enough to give them the degree of moisture control they really need to provide consistent quality cast products.

This also applies to the standard actual moisture test. Its use is primarily for determining the actual moisture of research batches of sand, or of sand as is delivered to the molders at the time of molding. The Actual or Standard Moisture Test is, in reality, a check on the Compensated Moisture Test.

In a continuous foundry operation sand conditions are checked on an hourly basis and adjustments made to the mixture (either sand, bonding material and/or water are added) depending on the basic standard test results.

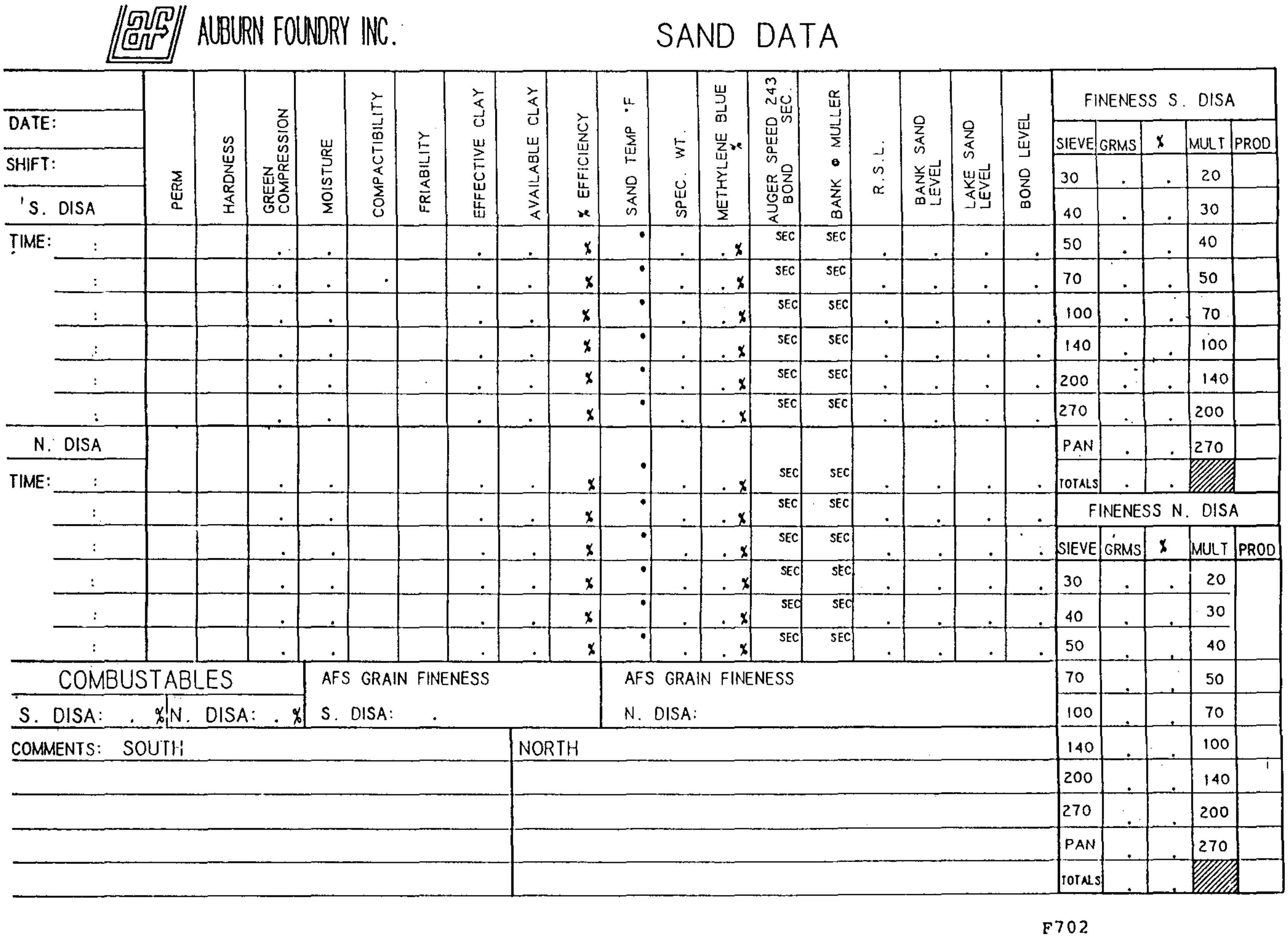
ADVANCING TECHNOLOGY TRANSFER

AUBURN FOUNDRY, INC.
AUBURN, IN

* Disamatic Molding System
* Sand Testing Laboratory
* Sand Data Collection Sheet
* SPC Sand Data Evaluation
    Daily
    Monthly Summary

AUBURN FOUNDRY GROUP EUROPE
EISENBERG, GERMANY

* Disamatic Molding System
* Sand Testing Laboratory
* Sand Data Collection Sheet
* SPC Sand Data Evaluation
    Daily
    Monthly Summary



AUBURN FOUNDRY INC.

SAND DATA

	PERM	HARDNESS	GREEN COMPRESSION	MOISTURE	COMPACTIBILITY	FRIABILITY	EFFECTIVE CLAY	AVAILABLE CLAY	% EFFICIENCY	SAND TEMP °F	SPEC. WT.	METHYLENE BLUE %	AUGER SPEED 243 SEC. BOND	BANK @ MULLER	R.S.L.	BANK SAND LEVEL	LAKE SAND LEVEL	BOND LEVEL
DATE:																		
SHIFT:																		
S. DISA																		
TIME: :			.	.			.	.	%	.	.	. %	SEC	SEC	.	.	.	.
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N. DISA																		
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FINENESS S. DISA

SIEVE	GRMS	%	MULT	PROD
30	.	.	20	
40	.	.	30	
50	.	.	40	
70	.	.	50	
100	.	.	70	
140	.	.	100	
200	.	.	140	
270	.	.	200	
PAN	.	.	270	
TOTALS	.	.	▨	

FINENESS N. DISA

SIEVE	GRMS	%	MULT	PROD
30	.	.	20	
40	.	.	30	
50	.	.	40	
70	.	.	50	
100	.	.	70	
140	.	.	100	
200	.	.	140	
270	.	.	200	
PAN	.	.	270	
TOTALS	.	.	▨	

COMBUSTABLES		AFS GRAIN FINENESS	AFS GRAIN FINENESS
S. DISA: . %	N. DISA: . %	S. DISA: .	N. DISA:

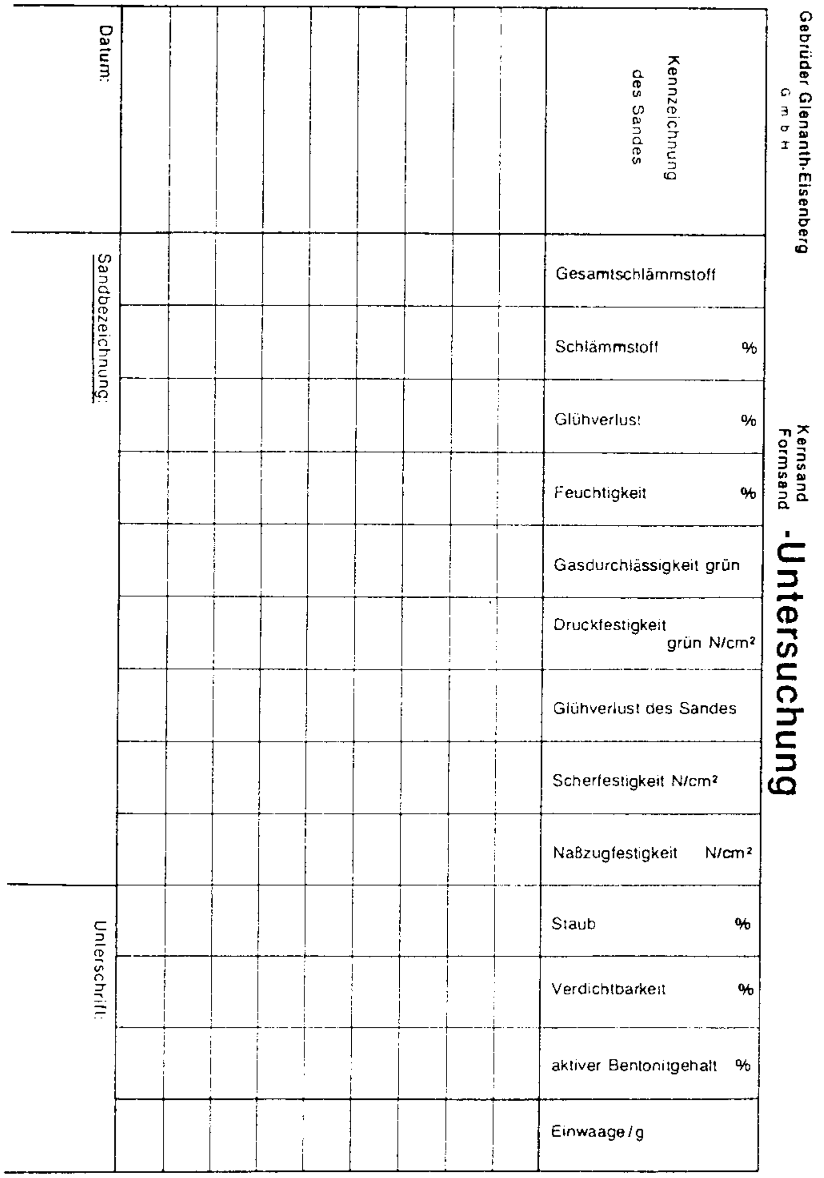
COMMENTS: SOUTH

NORTH

F702

SAND DATA COLLECTION SHEET
FROM SAND LABORATORY
AUBURN, INDIANA

SAND DATA COLLECTION SHEET
FROM SAND LABORATORY
EISENBERG, GERMANY



Gebrüder Glenanth-Eisenberg
G m b H

Kernsand
Formsand -Untersuchung

Datum:

Sandbezeichnung:

Unterschrift:

Kennzeichnung des Sandes	
Gesamtschlämmstoff	Total clay
Schlämmstoff %	Clay content %
Glühverlust %	Loss on ignition %
Feuchtigkeit %	Moisture content %
Gasdurchlässigkeit grün	Permeability of green sand
Druckfestigkeit grün N/cm²	Compressive green strength
Glühverlust des Sandes	Friability
Scherfestigkeit N/cm²	Shear green strength
Naßzugfestigkeit N/cm²	Wet tensile strength
Staub %	Dust content %
Verdichtbarkeit %	Compactability %
aktiver Bentonitgehalt %	Effective day content %
Einwaage/g	Total weight

Phase I: Testing for Moisture Content:

The Moisture Teller produces a rapidly moving stream of hot air which is directed at a weighed sample of the material to be checked. The hot air dries out the sample in a few minutes and after reweighing, only a simple calculation is required to determine the percent loss or percent moisture in the original material.

To determine moisture with the No. 276 Moisture Teller, there are three steps to be followed:

1. When the No. 250 Laboratory Balance is used, place one of the tared (weighed) pans on the right-hand balance pan and the counter-weight on the left, with the slide weight at zero. Check to see that it balances and, if not, adjust the knurled nuts accordingly. Add the 50 gram weight to the left-hand balance pan, and then quickly place the material under test in the moisture pan until a balance is obtained. (Any undue delay here will cause loss of moisture during weighing and will lead to low results.) Distribute material evenly in the pan.

2. Insert pan into Moisture Teller by depressing rubber button on the pan holder and sliding pan in so that it sits evenly in the holder. Release pan holder and see that the pan fits snugly around its top edge against the body of the Moisture Teller. Start the Moisture Teller by turning the timer pointer to the drying time desired.

3. When the timer turns off the Moisture Teller, remove the pan by means of the lifter and replace it on the right-hand balance pan. Move the 1 to 10 gram slide weight to the right until a balance is obtained. The reading of the slide weight scale multiplied by 2 equals the percent moisture. If the sample contains more than 20 percent moisture, a 10 gram weight must be placed with sample pan on the right-hand pan and added to the slide weight reading.

OR

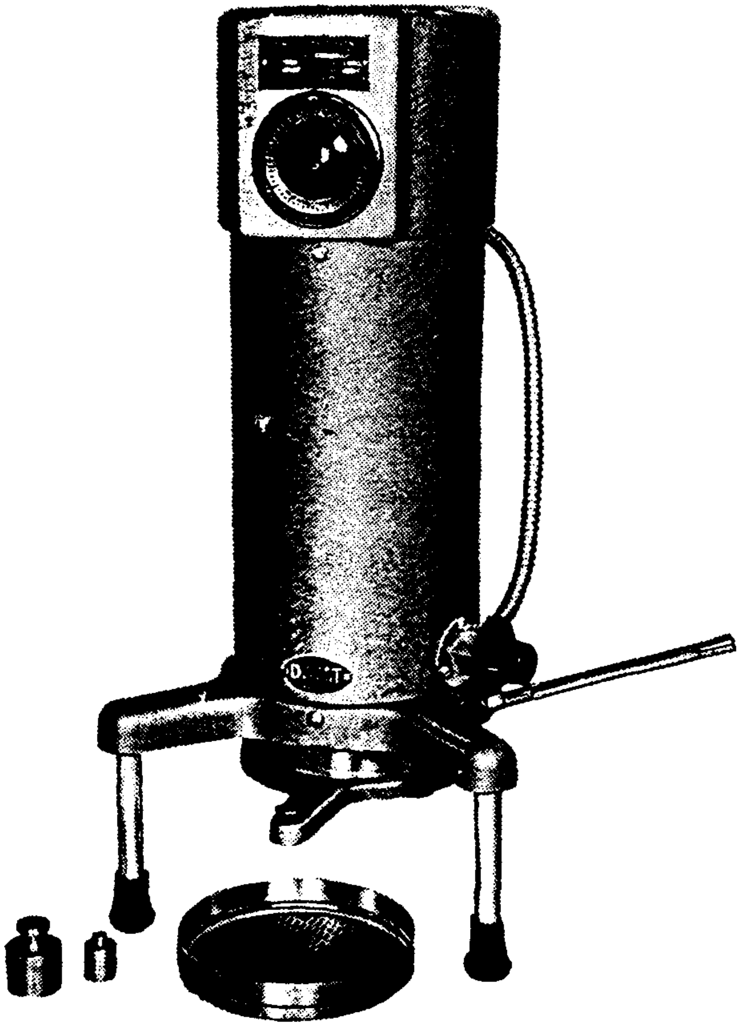
Determine percentage:

$$\% \text{ Moisture} = \frac{\text{moist weight-dried weight} \times 100}{\text{Moist Weight}}$$

NOTE: Clean the sample pan after each use.

The counterweight may wear with continued use and the pans may acquire adhering dirt, so it is advisable to check the pans against the counterweight periodically.

Moisture teller.



Phase II: Class Tour of local foundry to observe the total foundry process with emphasis on sand testing and control

Sands for foundry operations are usually a mechanical mixture containing as much as 98% silica sand, plus clay, carbonaceous material, and water. Fireclay, western and southern bentonite are used usually in combination to bond the sand grains. The carbonaceous material may be ground bituminous coal (sea coal), cellulose (cereal and wood flour) or any other special compounded material either natural or synthetic.

Most companies after years of experience have developed the "correct sand specification" for their product line.

Automatic molding systems are common in most of today's foundry operation. An example of one such system is the DISAMATIC method (this is a flaskless vertical parted green sand molding process).

auburn foundry, inc.

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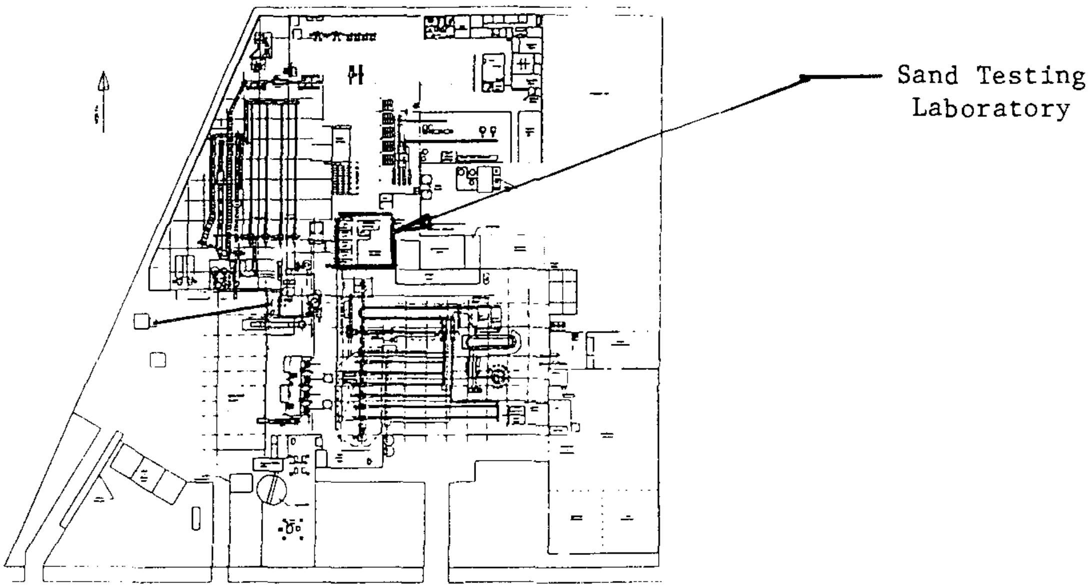


DISAMATIC Molding Machine (DMM)

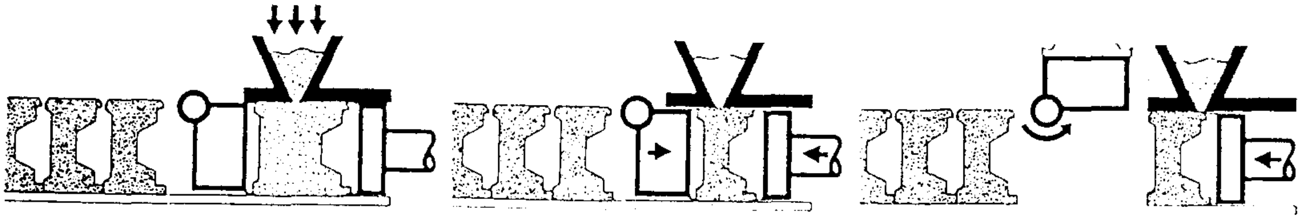
MOLDING PRINCIPLE AND MACHINE DESIGN FEATURES

The 2070 DISAMATIC is a shot-squeeze molding machine producing flaskless, vertically parted sand molds.

The essential components for the production of the molds are:

- a molding chamber with fixed bottom, top and side walls,
- movable pattern plates,
- a sand hopper,
- a sand shot arrangement, and
- a hydraulic system providing the force for squeezing and transporting the molds.

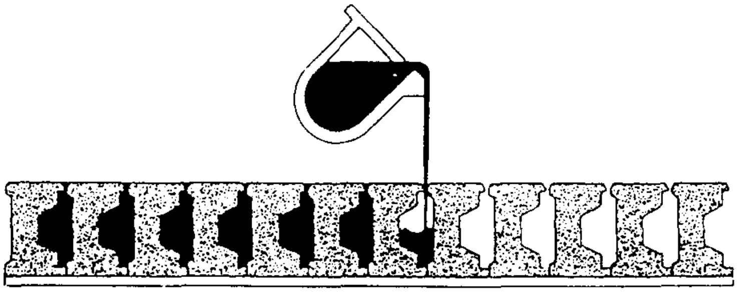
The principle of the DISAMATIC molding method.



Filling the molding chamber with sand

Squeezing the mold

Stripping the pattern and transporting the molds



Define the following terms as they apply to sand testing in today's foundry operations:

Sand ______________________________

____

Clay ______________________________

____

Moisture Content ______________________________

____

Grain Size ______________________________

____

Permeability ______________________________

____

Mold Hardness ______________________________

____

Green Compression ______________________________

____

Friability ______________________________

____

Sand Temperature ______________________________

____

Compactibility ______________________________

____

Naturally Bonded Sands ______________________________

____

Synthetic Sands ______________________________

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REFERENCES:

1. Dansk Industri Snydikat, Disa Foundry Technology, Printed in Denmark, 1989.

2. Diehm, Michael, Product Development Engineer, Auburn Foundry, Auburn, Indiana, August 26, 1994, Plant Tour, Review of Sand Testing Laboratory and Collection of Sand Test Data.

3. Doyle, Lawrence E., Manufacturing Processes and Materials for Engineers, 3rd Edition, Prentice-Hall, Inc., 1985.

4. Heine, W. Richard, Principles of Metal Casting, McGraw-Hill Book Company, 1967.

5. Lyman, Taylor (Editor), Metals Handbook Vol. 5, Forging and Casting, 8th Edition, American Society for Metals, 1970.

6. Schwan, Hermann, Production Manager of Foundry Operation Eisenberg, Germany, August 11, 1994, Plant Tour, Review of Sand Testing Laboratory, and Collection of Sand Test Data.

OBSERVING AND MODELING CREEP BEHAVIOR IN WOOD

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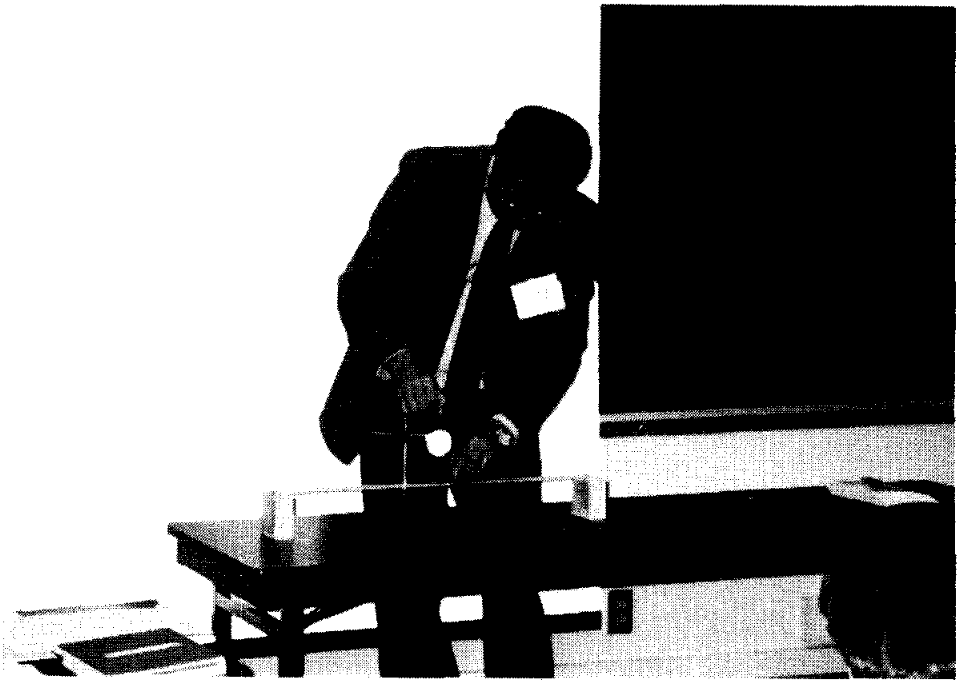

Observing and Modeling Creep Behavior in Wood

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University of Idaho
Moscow, Idaho

Key Words: creep, creep models, load duration, wood properties

Prerequisite Knowledge: Some knowledge of elastic properties of materials or beam theory is helpful but not necessary. A basic understanding of logarithms is necessary to perform the numerical analysis and curve fitting exercises.

Objective: To observe the creep phenomenon in a wood beam placed under constant load. This demonstrates the need for wood engineers to adjust allowable design stresses for duration of load. A second objective is to use experimental data to evaluate various numerical methods for creep analysis.

Equipment:

1. clear, straight-grained wood beam (measuring approximately 1/8" x 1-3/4" x 30")
2. weight (approximately 1 lb)
3. blocks sufficient to support beam ends about 3-4 inches above table
4. stop watch
5. dial gage with support arm
6. log-log graph paper

Introduction:

Creep effects under mechanical stress are observed in most solid materials. For wood-based structural components, creep and its effect on strength reduction must be accounted for by considering the "duration of load."

For example, the National Design Specification (NDS) for Wood Construction (NFPA 1991) provides allowable stress adjustment factors for various types of loads — wind, snow, seismic, and permanent loads, Depending on whether the expected design loads are in place for less than or more than 10 years (normal duration), allowable design stresses may be increased or reduced respectively (Figure 1). In addition, ASTM procedures for testing wood samples (ASTM 1991) include specific time limits for reaching ultimate stress, as a way to minimize the effect of loading rate on stress—strain relationships.

This experiment illustrates the need for duration of load adjustment factors by measuring creep in a simply-supported wood beam and plotting the results on a log-scale. In addition, mathematical creep models that have been put forth by various researchers are presented, allowing the opportunity for their assessment by fitting the experimental data to the various models.

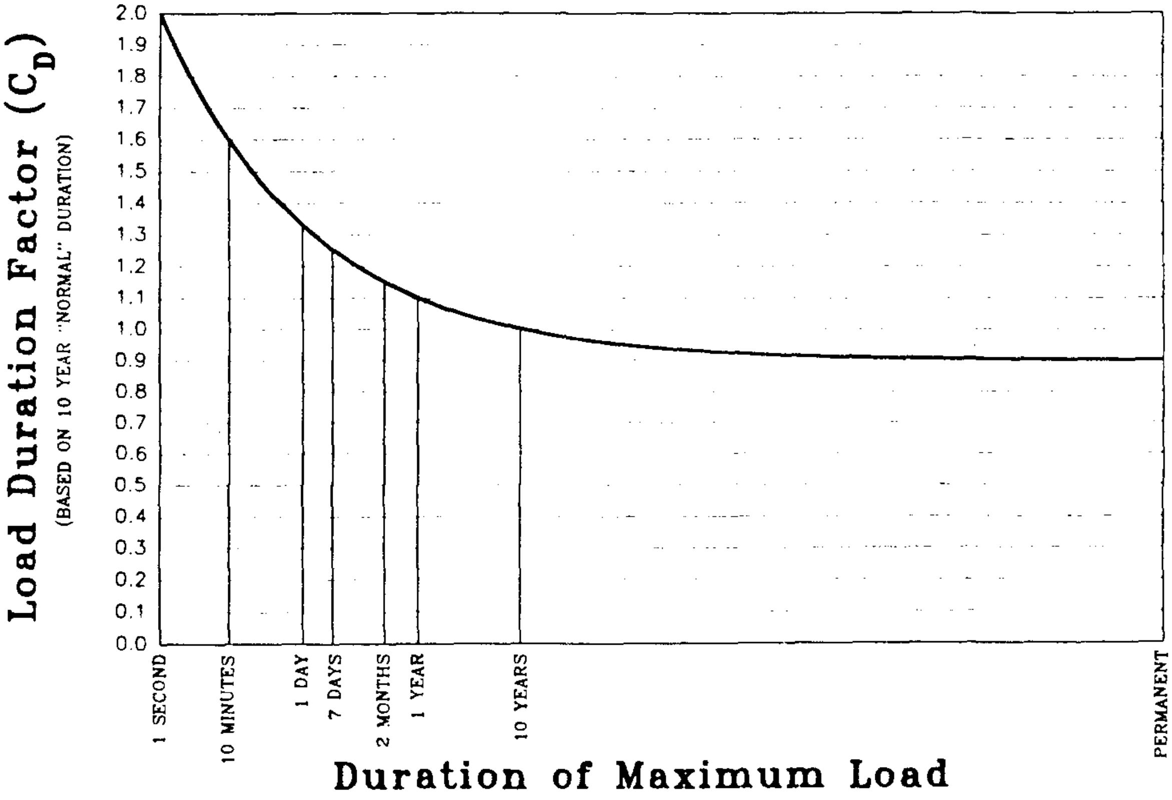

Figure 1. Load duration factors, C_D, for various load durations (NFPA 1991).

Procedure:

The phenomenon of creep can be observed by loading a wood beam, such as that described above in the equipment list, about its weak axis, and measuring deflection over time. As illustrated below (Figure 2), the beam is supported on blocks, allowing about 1 inch at each end to rest on the support blocks. The weight is positioned at mid span, and deflection is recorded over time with the dial gauge.

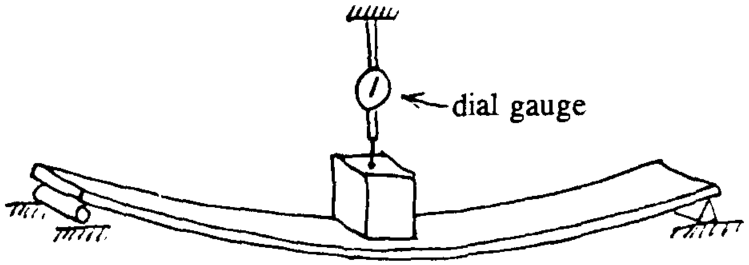

Figure 2. Test set-up.

The dial gauge can be zeroed as soon as the load is in place, thus ignoring initial deflection at time zero. That way, subsequent measurements of defection will pertain only to deflection due to creep (Δ_{cr}). The deflection data can be recorded on a table similar to Table 1. The suggested time intervals are useful when plotting the results on a logarithmic scale.

Time (min.)	½	1	2	5	10	20	30	45	60
Δ_{cr} (in.)	.003	.004	.005	.007	.013	.017	.018	.019	.020

Table 1. Data collection table with experimental data added.

Analysis of Data:

Many empirical creep equations have been developed by researchers and are available in various publications (Bodig and Jayne 1982, Conway 1967, Cristwell and Vanderbuilt 1982). A few of these equations are included in Table 2.

Name	Creep Range	Equation	Constants
Parabolic	primary	$\Delta_{cr} = at^m$	a,m
Logarithmic	primary	$\Delta_{cr} = a + b \log t$	a,b
Polynomial	primary and secondary	$\Delta_{cr} = a + bt^{1/n} + ct^{2/n} + dt^{3/n}$	a,b,c,d,n
Hyperbolic sine	primary and secondary	$\Delta_{cr} = a + b \sinh ct^m$	a,b,c,m (m=1/3 usually)
deLacombe	primary, secondary and part of tertiary	$\Delta_{cr} = at^m + bt^n$	a,b,m,n

Table 2. Some commonly used empirical creep equations.

One of the relatively simple equations that can be fit to the data that is collected is the parabolic form:

$$\Delta_{cr}=a(time)^m \tag{1}$$

The constants can be determined by plotting the data on log-log paper and fitting a straight line. Simple statistical techniques, such as least-squares curve fitting, can be used to find the best fit. Constant *a* is the creep deflection at 1 time unit. The slope of the fitted line is the constant *m*.

Another form of Equation 1 can be developed by taking logarithms:

$$\log(\Delta_{cr}) = \log(a) + m\log(time) \tag{2}$$

By plotting log (Δ_{cr}) as a function of log (time) the parameters a and m can be determined. Again, because the transformed relationship is linear, simple straight-line fitting can be used to obtain values for a and m. As shown in Figure 3, which was developed with data taken from the test set-up as described in the procedure, the slope of the plot is m and the intercept of the straight line is log a. In this case, $m = 0.4298$ and log $a = -2.4$. Thus, $a = 0.00398$ for use in Equation 1. Figure 3 was produced using a spreadsheet with a regression function, and using the logarithims of the data shown in Table 1 as input. The R^2 for this regression analysis was 0.9365.

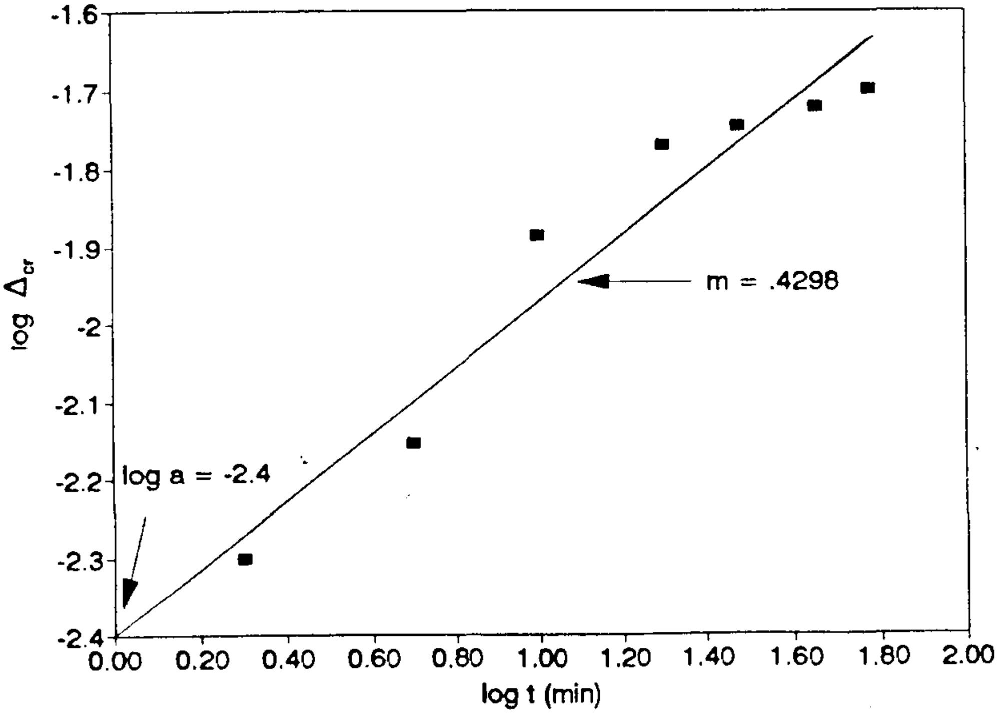

Figure 3. Relationship between log (Δ_{cr}) and log (time) for a spruce beam.

Notes to the Instructor:

1. The dimensions of the bending member or the magnitude of the weight are not critical of themselves, but they should be designed to create an equivalent bending stress of about 1500 psi. The combination illustrated in this experiment results in a bending stress just over 1400 psi.

To determine bending stress in a simply-supported beam with a single, concentrated load as illustrated, use the following formula (derived from the flexure formula):

$$\sigma = 1.5\frac{PL}{bd^2} \tag{3}$$

where:

σ = stress (psi)
P = load (pounds)
L = span length (inches)
b = width of beam (inches)
d = depth of beam (inches)

2. Creep is affected by several factors, including the stress level on the beam, the temperature, and the moisture content of the wood. Additional creep tests can be conducted, for comparison, by altering the load level or condition of the wood. Plywood might offer another method of comparison, since the grain direction of test samples can be easily oriented when sawing the samples.

3. While the NDS provides a single load duration factor (C_D) relationship, it may be interesting to compare creep effects for different species of wood. For example, hard maple has a much higher density than does spruce. Be sure that all other factors (size of members, moisture content, etc.) remain constant.

References:

American Society for Testing and Materials: Standard methods of testing small clear specimens of timber. ASTM D 143-83. ASTM, Philadelphia, Pennsylvania, 1991.

Bodig, J. and B.A. Jayne: *Mechanics of Wood and Composites*. Van Nostrand Reinhold Co., New York, 1982.

Conway, J.B.: *Numerical Methods for Creep and Rupture Analysis*. Gordon and Breach, Science Publishers, Inc., New York, 1967.

Criswell, M.E. and M.D. Vanderbuilt: Laboratory testing of wood and structural wood products. *in: Wood as a Structural Material*. Dietz, A.G.H., E.L. Schaffer, and D.S. Gromala, eds., Educational Modules for Materials Science and Engineering (EMMSE) Project, Materials Research Laboratory, The Pennsylvania State University, University Park, Pa, 1982.

National Forest Products Association: *National Design Specification for Wood Construction*. NFPA, Washington, DC, 1991.

THE DEVELOPMENT OF MECHANICAL STRENGTH IN A CERAMIC MATERIAL DURING FIRING

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DEVELOPMENT OF MECHANICAL STRENGTH IN A CERAMIC MATERIAL FIRED AT SEVERAL DIFFERENT TEMPERATURES

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Richland High School, Richland, WA

Key Words: Ceramics, Extrusion, Modulus of Rupture, Strength Testing, Brittle Materials

Prerequisite Knowledge: This experiment was designed for high school students who have a very limited background in ceramic materials, but could be used at the college level.

Objective: To teach students about: 1) the development of mechanical strength in a ceramic material as a result of the firing process, and 2) about testing methods used for brittle materials.

Procedure: To begin, introduce the concept of brittleness, which is failure at very low values of mechanical strain. Although ceramic materials can be stronger than some metals, the measured strain at failure is very low, typically 0.01 to 0.1% strain. This is because the typically ionically bonded ceramic cannot relieve the high stresses at inherent flaws by flow, as usually occurs in metals and polymers. Cracks remain sharp and propagate through the material at high speed. The ceramic article tends to break catastrophically. There are exceptions to this behavior, with certain ceramic composites or new ceramics processed for toughness, but the generalization holds true for glasses and traditional clay-based ceramics.

In this experiment, we will prepare clay-based ceramic bars of uniform circular cross section by extrusion. Then we will fire the bars at temperatures high enough to achieve high strength by development of a glassy bonding phase. Finally, the bars will be stressed to failure in a simple testing machine. This will quantify the increase in strength as a function of firing temperature.

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* Pacific Northwest Laboratory is operated by Battelle Memorial Institute for the U.S. Department of Energy under Contract DE-AC06-76RLO 1830.

First, purchase some low-firing clay suitable for extrusion from your local hobby ceramic supplier. Such a clay should be free from large particles which would cause tears during extrusion. (The clay we used is of the same composition as the slip used to make ceramic bells for a presentation in NEW:93 [Bunnell and Piippo, 1993]). Since the clay will be extruded, ask the supplier to make the clay slightly higher in water content than would be used with a potter's wheel. Use a simple, inexpensive hand-operated extruder to extrude the clay to a uniform circular cross-section about 2 cm in diameter. If the extruder die has multiple holes of different sizes, construct a cover for the sizes not desired, to prevent extrusion through those holes. Cut the clay as it emerges from the extrusion die into approximately 0.5 m lengths, straighten them as much as possible by rolling on a bench top, and allow them to air dry overnight at room temperature. Then cut each clay bar into segments about 20 cm long by hacksawing about 1/3 through and carefully breaking it. Scratch the intended firing temperatures near one end of each bar. A minimum of thirty bars are required, which allows for testing of five specimens at each of five firing temperatures, plus five bars to be tested without firing them. About ten extra bars should be produced in case of mistakes or accidental breakage. For our clay composition, we used the following firing temperatures: 650, 750 850, 950 and 1050 C. Heat the specimens to the firing temperature in about four hours, hold at that temperature for 1.5 hours, and allow them to furnace cool.[1] Specimens that have been dried but not fired should also be tested for comparison.

In order to test the strength of the ceramic specimens, several methods were considered in terms of simplicity and validity of results. For instance, the direct application of tensile stresses to a ceramic specimen is difficult. In order to obtain valid results, tensile specimens must experience no bending loads. Another method, diametral compression of a right circular cylinder is good in that the specimen is inherently self-aligning, but trimming the specimen faces parallel is difficult and the force required to cause failure probably exceeds our measuring

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[1] Depending on the size of the clay body being fired, steam formation could cause specimens to explode during firing if the temperature increases too quickly. We did not experience any difficulty at the specimen diameter used, but the problem could be avoided by drying the specimens overnight at a temperature of about 90°C.

capability. Three-point bend testing of a uniform cross-section is a reasonable approach, and the specimen is self-aligning if circular in cross-section.

For our testing, we built and used the simple testing machine shown in Fig. 1 (cost less than \$50).[2] Of course, any universal testing machine could be used. The failure of ceramics is relatively insensitive to strain-rate effects, which permits us to apply the load at an uncontrolled rate without introducing major error. Loading the specimens in three-point bending also permits foreknowledge of the failure location (the center of the top of the test bar), and any obvious surface flaws can be simply placed on the compression side of the specimens so that they have a smaller influence on the results. Results in which the specimen did not fail near the center of the test bar where stress is known should be ignored.

The size and loading conditions for specimens should be chosen to minimize stored energy in the test machine and specimen, so that fragments will not be thrown when the specimen fractures. Also, the test setup we used would tend to throw fragments downward rather than outward. As long as three-point bending is used and the breaking force is less than about 1000 N, a scatter shield is probably unnecessary. Perform a dry-run of the experiment, following all steps and checking for unexpected safety hazards. If it appears that fragments could be thrown, the specimens could be tested while contained in polyethylene bags. During testing with our machine, one person operated the jack and a second person read the force gauge.

During testing of the specimens, only the specimen diameter and peak load are measured. When testing is complete, use these values in the equation for the maximum tensile stress sustained by the bar:

$$S = PL/\pi r^3 \tag{1}$$

In the above equation, S is the maximum stress on the tensile side of the specimen (Pa), P is

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[2] The design for this testing machine was adapted from one built by Andy Nydam, a high school Materials Science and Technology instructor at River Ridge High School in Lacey, WA.

the breaking force (N), L is the distance between the two outer load points (m), and r is the specimen radius (m). The maximum stress calculated by the above equation of traditionally called the modulus of rupture in the literature. The stress values for any given specimen condition will show scatter, which is typical for ceramic materials. (The fabrication methods used for ceramics tend to leave cracklike artifacts, unlike the pouring of a molten metal.) Average the strength values and calculate the standard deviation for each group of specimens fired at a given temperature.

A plot of our results is shown in Figure 2. Note that the strength of the fired ceramic increased at an accelerating rate with firing temperature. The strength increase is correlated with the development of a glassy bonding phase at temperatures above about 800 C, and the amount of this bonding phase increases with temperature. (For a general discussion of ceramic materials and the firing process, see Flinn and Trojan, Ch. 7, or Jacobs and Kilduff, Module 7.) The ceramic thus becomes stronger as it is fired to higher temperature. If the clay we used is heated another 100 C to 1150 degrees C, the glassy phase is sufficiently fluid and present at a high enough volume percentage that the ceramic will deform or slump. As shown in Figure 2, the standard deviation of specimen strength tends to increase with increasing strength. However, the scatter in the data is no greater than about 20%, which is in the expected range for ceramic materials fabricated by unsophisticated methods. Careful measurements will reveal that the cross section of each rod is not quite round, which could be a major source of error since the radius is cubed in the equation used to calculate fracture stress. If the diameter is measured at one point in the center and that point is marked, the specimen could be oriented so that load is applied parallel to the measured diameter.

Note also that the values of breaking stress in Figure 2 are very high. This is an artifact of testing by the three-point bend method, and is caused by the very small volume of material stressed to the calculated maximum level. Bend testing generally overestimates the true tensile stress as determined in a uniaxial tension test, by a factor of about three. This bias is of no consequence for the purposes of this experiment, since failure stresses are used only for comparison purposes.

Sample Data Sheets: None necessary.

Instructor Notes: There are many possible variations of this experiment. For instance, specimens could be fired for different lengths of time at the higher temperatures (above 800 C) to study strength development as the glass has more time to form and flow. Intentional defects could be introduced into the specimens, and oriented on either the tensile or the compressive faces. The effect of defects will be much more evident on the tensile face. Round ceramic rods subjected to a thermal shock test such as described in (Bunnell 1991) could be tested, and the decrease in strength with thermal shock damage could be measured.

References:

1. Flinn, R. A.; and Trojan, P. K.: *Engineering Materials and Their Applications, Third Ed.*, Houghton Mifflin Co., 1986.

2. Jacobs, J. A.; and Kilduff, T. F.: *Engineering Materials Technology, Second Ed.*, Prentice-Hall, 1994.

3. Bunnell, L. R.: *Thermal Shock of Ceramic Materials, presented at National Educators' Workshop*, Update 1991, NASA Conference Publication 3151, 1992.

4. Bunnell, L. R.; and Piippo, S.W.: *Property Changes During Firing of a Typical Porcelain Ceramic*, presented at National Educators' Workshop: Update 1993, NASA-Langley, 1993.

Sources of Supplies:

1. EM210 clay composition, made from 50% Talc, 50% Ball Clay, available from Laguna Clay Co., City of Industry, CA 91746

2. We used a Standard Clay Hand Extruder, made by North Star Equipment, PO Box 189, Cheney, WA 99004.

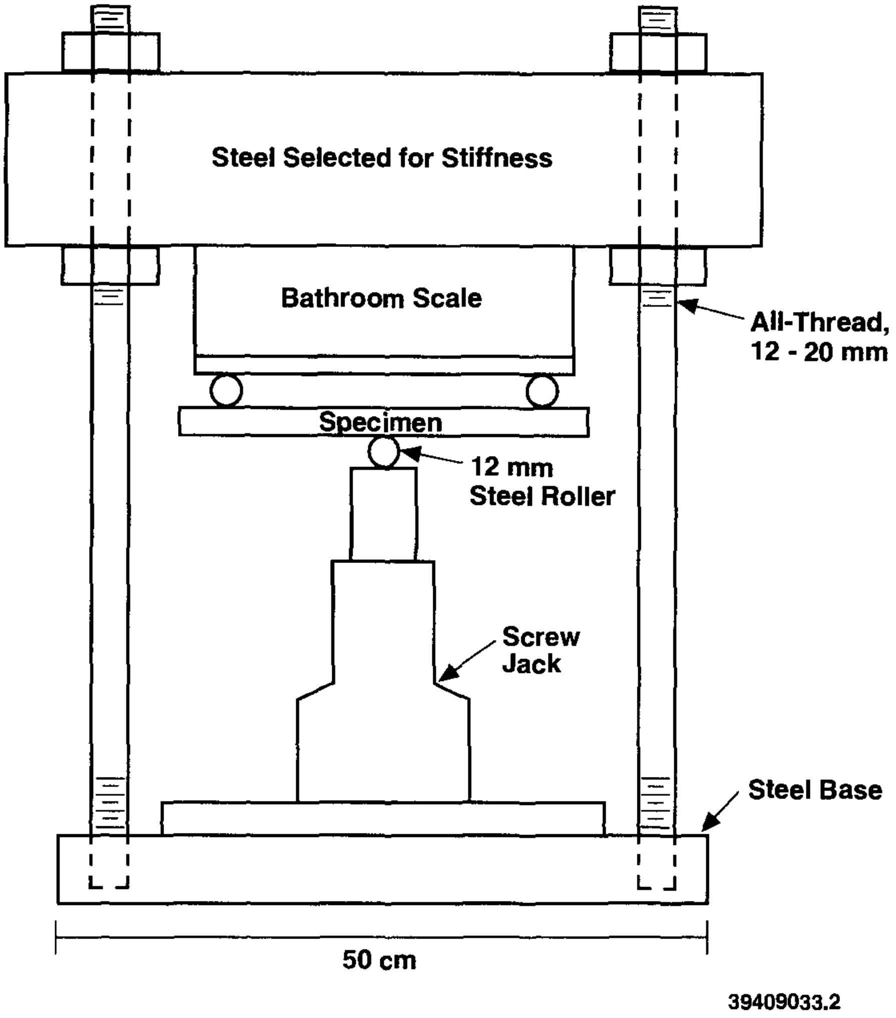

Figure 1. Testing machine used to load ceramic test bars in three point bending.

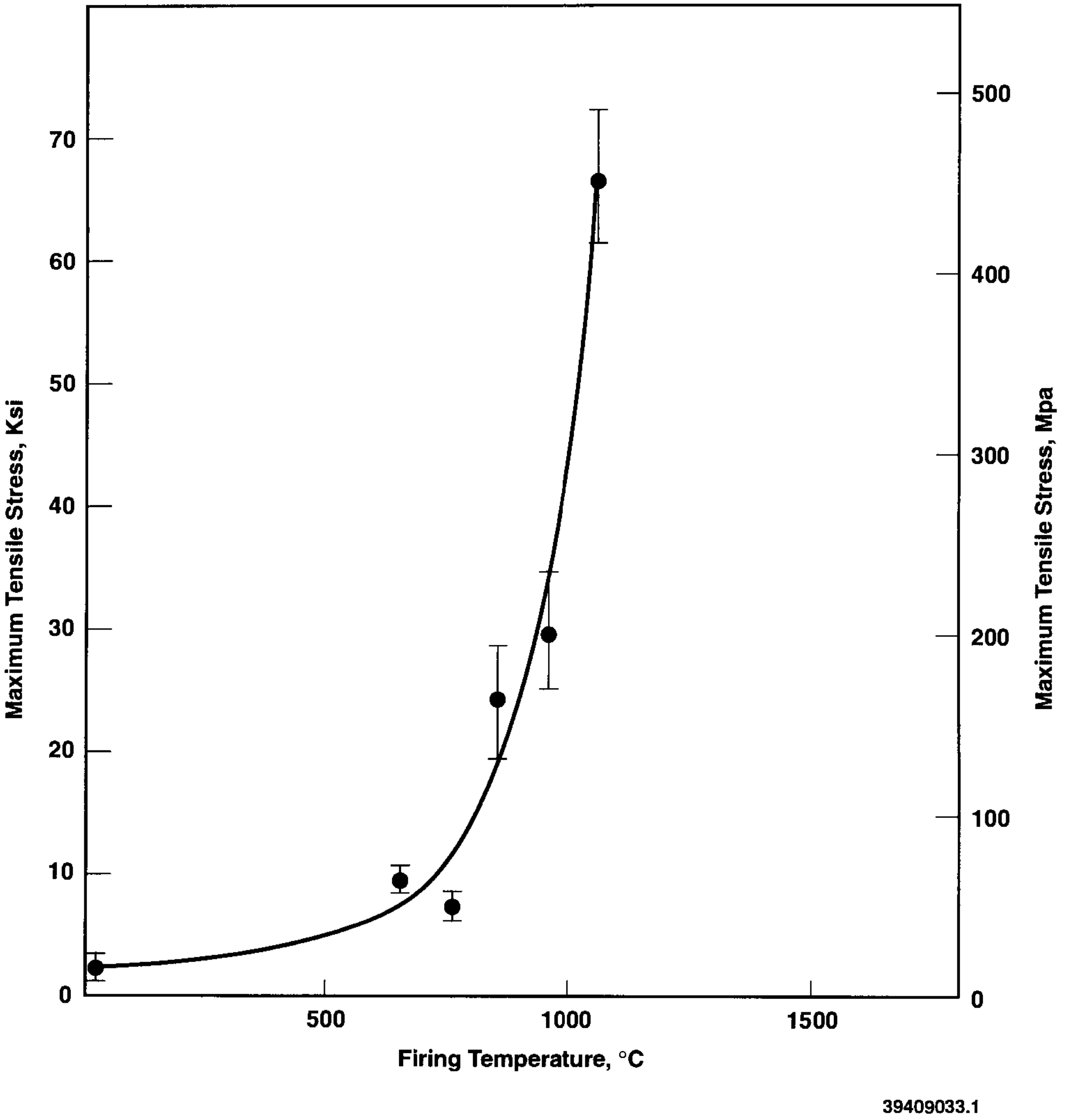

Figure 2. Strength of ceramic bars, as a function of firing temperature.

WATER DROP TEST FOR SILVER MIGRATION

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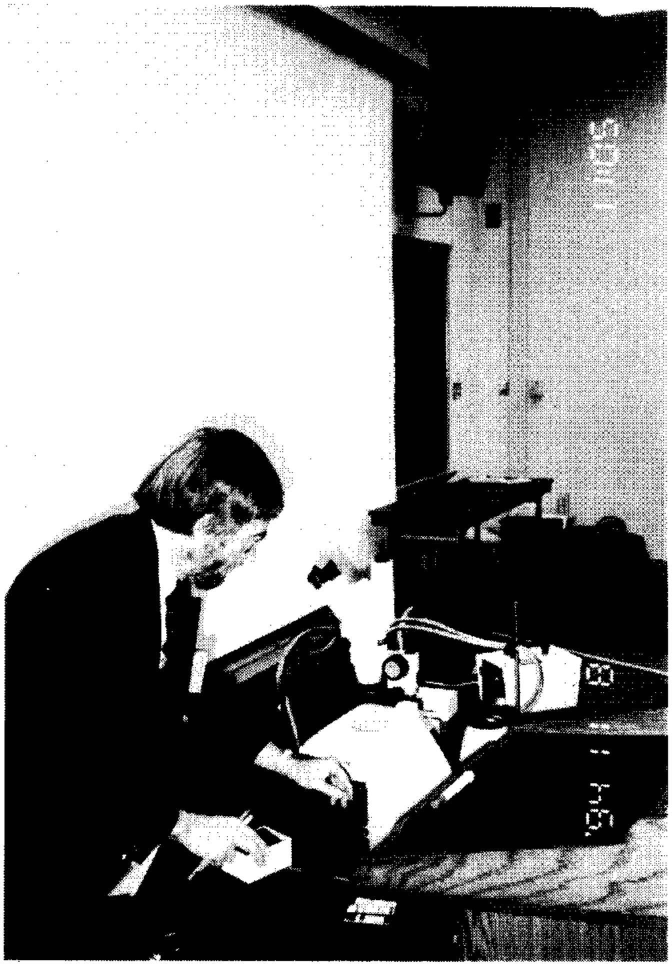

WATER DROP TEST FOR SILVER MIGRATION

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Key Words: Silver migration, thick film, dendrite, electrolyte

Prerequisite knowledge: Knowledge of precious metals and familiarity with electronic circuits is helpful.

Objective: To observe silver migration in a microcircuit application. To measure the effectiveness of a silver alloy for reducing silver migration.

Equipment:

1. 9 and 1.5 volt batteries
2. microscope, 10 to 30X
3. voltmeter
4. two electrical leads each with an alligator clip and probe tip
5. thick film test patterns with silver conductors
6. thick film test patterns with silver alloy conductors
7. DI water
8. stop watch
9. eye dropper
10. beaker, 50 ml

Introduction:

At room temperature silver has the highest electrical conductivity of any metal, but the high cost of silver prevents its use for many commercial applications. Silver does become economically feasible for microelectronic applications, because the quantity of silver required is small. One microelectronic technology that uses silver for circuit conductors is Thick Film. This technology uses the silk-screening process to produce miniature electronic circuits from conductor, resistor, and glass compositions. These materials are prepared as inks and a squeegee forces the ink through openings in a fine mesh screen and deposits it onto a substrate. Miniature circuits consisting of conductors, resistors, and capacitors can be created with this technology.

Unfortunately, a 100 percent silver conductor can not be used in all electrical applications. Silver can be easily oxidized or reduced. For example, silver in contact with vulcanized insulation can migrate and create conductor paths within the insulation which greatly reduces insulation resistance. Silver can also migrate in a thick film microcircuit and only requires the presence of moisture and a sufficient DC voltage gradient.

Moisture will form on most commercial circuits as a result of condensation from changing humidity and temperature conditions. Although the moisture may evaporate, the effect of silver migration is cumulative. The silver will begin migrating again with the next instance of condensation on the circuit. The final result can result in an electrical short between the two conductors.

This problem is an example of metallic corrosion, because a silver atom loses an electron and becomes a positive ion. An electrolyte consisting of water and positive silver ions is created. The silver ions acquire electrons at the negative electrode and metallic silver is deposited at the negative electrode. Thus, silver dendrites grow from the negative electrode to the positive electrode. Figure 1 shows dendrite growth between two conductor lines.

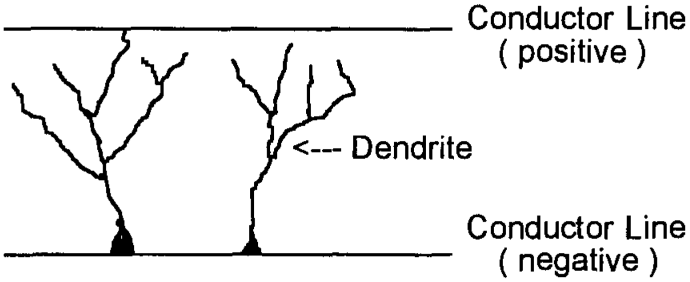

Figure 1. Dendrite Formation between conductor lines.

The distance between conductor lines, significantly affects silver migration in microcircuits. For thick film circuits the spacing between lines can be less than 0.2 mm, and this results in an electric field that is sufficient for migration. Increasing the voltage or decreasing the spacing between conductor lines increases the rate of silver migration.

Other metals such as copper, gold, platinum and aluminum do not experience migration with just water and a DC bias, because they do not readily form positive ions in water. However, these metals have other material problems or are too expensive for thick film circuits. To resolve the silver migration problem, material scientists alloy silver with other precious metals. The addition of 2% platinum or 15% palladium by weight will significantly reduce the rate of silver migration.

A common test that determines the resistance of a thick film conductor to silver migration is the water drop test. This is an accelerated test designed to generate dendrites in a short period of time. The test consists of placing a drop of water on two conductors that are < 0.2 mm apart. A DC voltage is applied at the test pads, and the time for the dendrites to bridge the gap is recorded. Figure 2 shows an example of a typical test pattern. The dendrites shown previously in Figure 1 will form in the migration area.

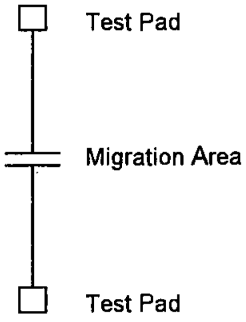

Figure 2. Migration test pattern.

Procedure:

1. Initial Preparation - Clean the 50 ml beaker and add ~ 20 ml of distilled water. Record the voltage of the battery and check lead continuity with the voltmeter.

2. All Silver Conductors - Attach the alligator clips to the test pads of the thick film substrate with the silver conductor. Place the sample under the microscope so that it is level and bring the migration area into focus. Add a drop of DI water over the migration area; make firm contact to the 1.5 volt battery with the tip of each probe; start the stop watch and with the microscope observe the test area for dendrite formation. Do all samples on the test pattern, and record the average time required to bridge the gap between the conductors.

3. Silver Alloy Conductors - Repeat the procedure in step 2 for the silver alloy samples.

4. Repeat steps 2 and 3 with a 9 volt battery and record all results in a table similar to the one below.

Table I. Sample Data Summary

Conductor Sample	Alloying Metal	DC Voltage, volts	Migration Time, sec	Comments
Group A	all Ag	1.5	15	
Group A	all Ag	9	5	Higher DC voltage
Group B	Ag/Pd	1.5	40	Pd alloying
Group B	Ag/Pd	9	20	Pd alloy & higher volt.

Notes to the instructor:

1. The thick film migration test patterns can be obtained from the author at Hocking College, 3301 Hocking Parkway, Nelsonville, OH 45764. Additional potential sources are:Engelhard Corp., Precious Metals Coating Group, 1 W. Central Ave., East Newark, NJ 07029 and E.I. DuPont, Microcircuit & Comp. Materials Div., Barley Mill Plaza, P30.2362, Wilmington, DE 19898.

2. The 9 volt battery may produce excessive generation of bubbles that can break up the dendrites and prevent them from bridging the gap. If this occurs you may have to use two 1.5 volt batteries in series or a 0 to 15 volt DC power supply.

3. The generation of bubbles results from the electrolysis of water and represents the production of hydrogen or oxygen gas depending on the pH of the water.

4. Keep the thick film substrates in a plastic bag to reduce tarnishing. Do not store them in a paper envelope, because it may contain sulfur.

5. Hold the sample by its edge to prevent salt contamination from your fingers. The salt will increase the rate of silver migration.

6. Be sure the beaker is clean and free of contaminants or faster migrations times may be incorrectly obtained.

7. The spacing between the conductors may vary for different sample lots. This could result in significantly different migration times for two lots of the same composition.

8. Samples can be reused by thoroughly cleaning the migration area with a rubber eraser.

References:

1. *McGraw-Hill Encyclopedia of Science and Technology*, sixth ed., McGraw-Hill Book Co. Vol. 16, 1987, pp. 420.

INEXPENSIVE EXPERIMENTS IN CREEP AND RELAXATION OF POLYMERS

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Inexpensive Experiments in Creep and Relaxation of Polymers

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Key Words: creep, relaxation, polymers, plastics

Prerequisite Knowledge: Some knowledge of solid mechanics/strength of materials/mechanical properties of materials.

Objective: To show that the mechanical properties of plastics are dependent on time. To alert the student to the design implications of putting plastics under constant loads.

Equipment:
1. Tensile specimens ("dogbones") of various plastics
2. Watch with second hand
3. Notebook to record data

For relaxation experiment
4. Tensile test machine

For creep experiment
5. Weights from bar-bell set
6. Small c-clamps
7. Metal wire
8. Ruler

Introduction:

Plastics are not often used for applications that must support a constant applied load. The reason for this is simple - under constant load, any plastic will deform if left for a long enough time. This will happen even at low loads and even at room temperature. Ceramics, wood, and most metals do not deform under the same circumstances. It is this type of deformation of plastics that limits the potential market for recycled plastic lumber. On the other hand, Higgins [1] points out the deformation of a plastic under its own weight is not so great that a plastic raincoat hanging in the closet winds up touching the floor.

The deformation of materials under conditions of constant load cannot be seen on a stress-strain curve, although it does explain why the speed of loading is important in plastics. To see it requires measurement of explicitly time-dependent behavior. There are two different time dependent behaviors that can be measured - creep and relaxation.

Creep occurs when a material under constant load deforms over time. Relaxation occurs when stress falls off in a material held at constant extension. In the lab creep and relaxation are measured separately. But in the real world, creep and relaxation typically occur together. Both are aspects of viscoelasticity, the overriding mechanical behavior of plastics [2;3].

A Creep Experiment:

There are commercial devices available to measure creep. These are costly, and if only one is available it takes a long time to get results. For student use, a simple experiment which measures many samples in parallel is more useful. The experiment shown here measures many samples simultaneously, at low cost.

Procedure:

Procure Tensile Dogbones of a Range of Plastics

Ours are made by students in the Plastics Engineering Technology program at Penn State-Behrend as a part of their studies in injection molding. They are thus available to us in large quantities at no cost. People who don't have this luxury may wish to cut specimens out of food containers (after eating the food, if you want our advice) or defunct shower curtains, or any available stock. It is possible to meet the ASTM standards by any of these methods [4;5]. It is always a good idea to meet the standard if possible, both to ensure a good test and to introduce students to the use of the relevant standards.

Choose, or make, dogbones of small enough cross-sections so that barbell weights result in stresses in the 1000 to 3500 psi range. ASTM D 638 type V are ideal [4].

It is best if all applied stresses are the same. This can be achieved by having uniform weights and cross-sections, or by varying the cross-sections to go with the available weights. An optimal experiment might be to use a 1/8 inch square cross section with a fifty pound weight, for a stress of 3200 psi.

Prepare a notebook page with two columns: time and length. The length should be the length of the gage area.

Attach c-clamps to top and bottom of specimen, on the wide parts of the specimens. Real grips would be better, but the c-clamps are close enough to the standard and much less expensive.

Hang the top end of the dogbone by a metal wire from a rigid support.

Hang the weight by a metal wire from the c-clamp attached to the bottom end of the dogbone.

Immediately measure the length of the specimen.

Continue to take measurements, every 15 sec. for the first five minutes, every five minutes for the first hour, at least once a day for as long as desired. The standard experiment takes the samples to creep rupture [5]. We found two weeks to be sufficient to demonstrate creep; even one day will give enough data for discussion.

Sample Results:

We used one sample each of five very different plastics. In order of increasing toughness, these were: polystyrene (PS), acrylonitrile-butadiene-styrene (ABS), polypropylene (PP), a nylon, and polycarbonate (PC). For this sample experiment no effort was made to make the stresses uniform. The samples were ASTM D 638 Type I, machined down to narrower cross-sections [4].
The sample dimensions, weights used and stresses are given in table 1.

Table 1: Sample Dimensions, Weights and Stresses

Material	Cross-Section area, sq. in.	Load, lb.	Applied Stress, psi
Polystyrene (PS)	0.017	25	1500
Acrylonitrile-butadiene-styrene (ABS)	0.030	30	1000
Polypropylene (PP)	0.015	15	1000
Nylon	0.027	40	1500
Polycarbonate(PC)	0.013	40	3000

The data are presented in Figure 1. The experiment was run for a little less than two weeks. These results are sufficient to establish that the experiment works, and that the experiment can be used to show that different plastics have different creep behaviors. Given that the samples were not all at the same initial stress, it is not easy to say anything further about the plastics. Creep rate is highly dependent on stress [1]. It is easy to recommend better practice when performing this experiment. Most of these recommendations are incorporated into the experimental procedure, above.

Further recommendations are: to do at least two samples of each material. One set of samples could be at 1000 psi, the other at 3000 psi. Separate plots could be made of:

1. All the samples at 1000 psi
2. All the samples at 3000 psi
3. A single material at 1000 psi and 3000 psi.

This would enable comparison of creep behavior across materials, and of the effect of increased stress on each material. If the experiment was done carefully enough, it should be possible to calculate the creep constants if the instructor so desired [7].

The Relaxation Experiment:

Unlike the creep experiment, this experiment has been run in the materials lab at Penn State-Behrend for five years, over at least 50 lab groups. It is thus well-established.

Procedure:

Procure Tensile Dogbones of a Range of Plastics

Use the same materials as in the creep experiment. Standard dogbones of any shape will work. Our dogbones are ASTM D 638 Type I, gage area 1/8" x 1/2", gage length 2.25 " [4]. Table 2 lists all of the materials we have used. ASTM practice [6] allows machined from existing materials; the samples do not have to be dogbones.

Table 2: Polymers used for Relaxation Experiment, with Approximate Failure Stresses

Material	Failure stress, psi	Type of failure
Elastomers	< 1600	Cannot support greater stresses
Polyethylenes	< 4800	Cannot support greater stresses
Polypropylene	< 4800	Cannot support greater stresses, or pulls apart
Recycled PP	Slightly less	
Acetates	< 4800	Brittle
Polystyrene	< 4800	Brittle
ABS	< 6000	Brittle or pulls apart
Nylons	< 8000	Pulls apart
Polycarbonate	> 9600	Pulls apart

Measure and record the specimens

Use a Tensile Tester

The ideal tester is accurate to one pound in a range of zero to one thousand pounds, for the gage cross section we use. This gives a stress range of zero to 16000 psi. Our tester has a maximum of 500 pounds. As is shown in table 2, this makes it incapable of breaking polycarbonate, but more than sufficient to break every other plastic we have used. The ideal tester would have computer data collection. The steps that follow assume hand collection of data.

Prepare a notebook page with two columns: time and force. The force is the raw data from the tensile tester.

Install a Specimen in the Tester

Increase the force until a stress of around 3000 psi is reached. Then stop the motion of the crossheads. A constant strain has now been applied, and the stress is expected to fall off. 200 lbs on our dogbone cross section gives a stress of 3200 psi. Generally elastomers cannot reach this stress. In all other polymers, this stress is less than the failure stress, but high enough that some interesting relaxation behavior should be seen.

Immediately Take a Measurement

Continue to take measurements, every 15 sec. for the first five minutes. Even three minutes gives enough data for useful results. If desired, the last sample can be left in the tester until the next lab meeting. The shape of the relaxation curve does not change, nor does it ever approach an asymptote. Stress just continues to fall. The longest time we have let a sample relax is two weeks.

Leaving the sample in the tester, increase the stress. We generally increase to 250 lbs (4000 psi) then 300 lbs (4800 psi) for each sample, and go on to 400 lbs (6400 psi) and 500 lbs (8000 psi) for those plastics which can support those loads.

In theory it may be better to use a new sample for each stress, but we have found that the differences between samples of the same batch of the same plastic are as large as the errors that may be introduced by just continuing to use the loaded sample. And using the loaded sample saves time. Repeat the procedure with each material.

Report:

The lab report requires the students to plot:
1. All the plastics at one stress, and
2. One plastic at three or more different stresses.

Figure 2 shows a sample result of stress relaxation for five plastics at one initial stress. The students are also required to calculate a relaxation time for all the materials. For example, the half-time of a material is the time it takes for the material to relax to half of the initial stress. As this is never reached in this experiment, the students are required to calculate the time it takes for the stress to fall to 95% of its initial value. For example, at an initial stress of 3200 psi, the time at which the stress reaches 3040 psi is reported. A table of these values for all plastics tested is reported by our students. This gives some measure of the relative relaxation resistance of the plastics. If the instructor so desired, the students could be asked to calculate the relaxation constant which Flinn and Trojan [8] and Shackleford [9] call the relaxation time, which is the time it would take for the stress to fall to 0.37 (= 1/e) of its initial value.

Discussion

This experiment is quite successful in showing that stress behavior of plastics is time-dependent. The students are asked to write one paragraph about the importance of this result for design; this has been a successful exercise for them.

Of the two properties, creep and relaxation, creep is more important. It is found in practically all introductory materials texts at every level - technology [1;2], design [7;8], materials science [9] and even physics [10]. In practice it is easier to picture a constant load application than a constant strain one. The students have been asked to explain the significance of relaxation for creep. This has not been very successful. It is easy enough to state that both are non-steady-state material responses. But for the purposes of actually seeing creep, it is necessary to perform a creep experiment.

Conclusions

Two experiments have been presented. One has been used for many lab sections with great success; the other is newly developed. Both should be useful for explaining some aspects of design with plastics.

In addition, they can be used as inexpensive analogues for metals experiments. Because of our proximity to a cheap source of plastic experimental pieces, we use plastic samples for much of our materials testing. This has proved quite useful for explaining the range of behaviors across various materials classes. There are brittle plastics, like polystyrene (PS). There are also tough plastics, like polycarbonate (PC). The tensile test results for PS look like those for a ceramic. The tensile test results for PC look remarkably like those for low carbon steel. In both cases the results for plastics are shifted down to lower stresses and out to higher strains. Similarly, plastics impact testing is much cheaper than metals impact testing. The same range of behaviors can be shown, including sensitivity to forming methods. Of course, plastics forming methods are not the same as heat treating.

Creep and relaxation in metals are high temperature phenomena, but room temperature is a high temperature for a polymer. All polymers melt or burn below 500 celsius [2]. Most, including all of those used here are destroyed below 300 celsius. Thus room temperature is above the rule of thumb temperature for creep and relaxation behavior in plastics. If one uses a rule of thumb of 1/3 of the melting point in degrees Kelvin as the onset of creep [7], a melting point of 300 celsius gives creep being important at any temperature above -80 degrees celsius. Thus plastics will always creep and relax in use.

This explains why plastics are not presently good for structural uses. Creep and relaxation will always lead to failure of plastics under constant loads.

References

1. Higgins, R.A.: *Properties of Engineering Materials.* Edward Arnold, London, 1977.
2. Jacobs, J.A.; and Kilduff, T.F.: *Engineering Materials Technology.* Prentice-Hall, Englewood Cliffs, NJ, 1985.
3. Young, R.J.: *Introduction to Polymers.* Chapman and Hall, London, 1981.
4. ASTM D 638 - 89: *Standard Test Method for Tensile Properties of Plastics.* American Society for Testing and Materials (ASTM), Philadelphia, 1990.
5. ASTM D 2990 - 77: *Standard Test Method for Tensile, Compressive, and Flexural Creep and Creep-Rupture of Plastics.* American Society for Testing and Materials (ASTM), Philadelphia, 1990.
6. ASTM D 2991 - 84: *Standard Practice for Testing Stress-Relaxation Plastics.* American Society for Testing and Materials (ASTM), Philadelphia, 1990.
7. Ashby, M.F.: *Materials Selection in Mechanical Design.* Pergamon Press, Oxford, 1992.
8. Flinn, R.A.; and Trojan, P.K.: *Engineering Materials and Their Applications*, 3rd ed. Houghton Mifflin Co., Boston, 1986.
9. Shackleford, J.F.: *Introduction to Materials Science for Engineers*, 2nd ed. MacMillan Publishing Co., New York, 1988.
10. Lovell, M.C.; Avery, A.J.; and Vernon, M.W.: *Physical Properties of Materials.* Van Nostrand Reinhold, New York, 1976.

Fig. 1:

Results of sample creep experiment

5 plastics, 1000 to 3000 psi

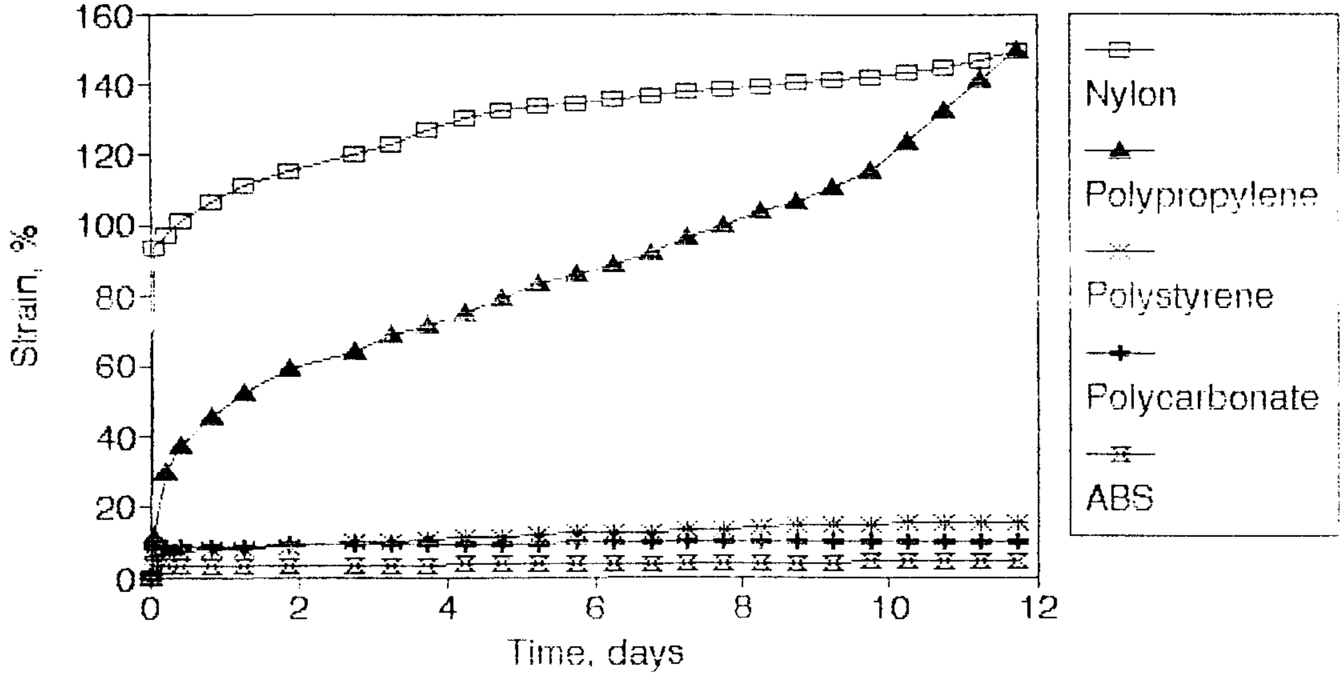
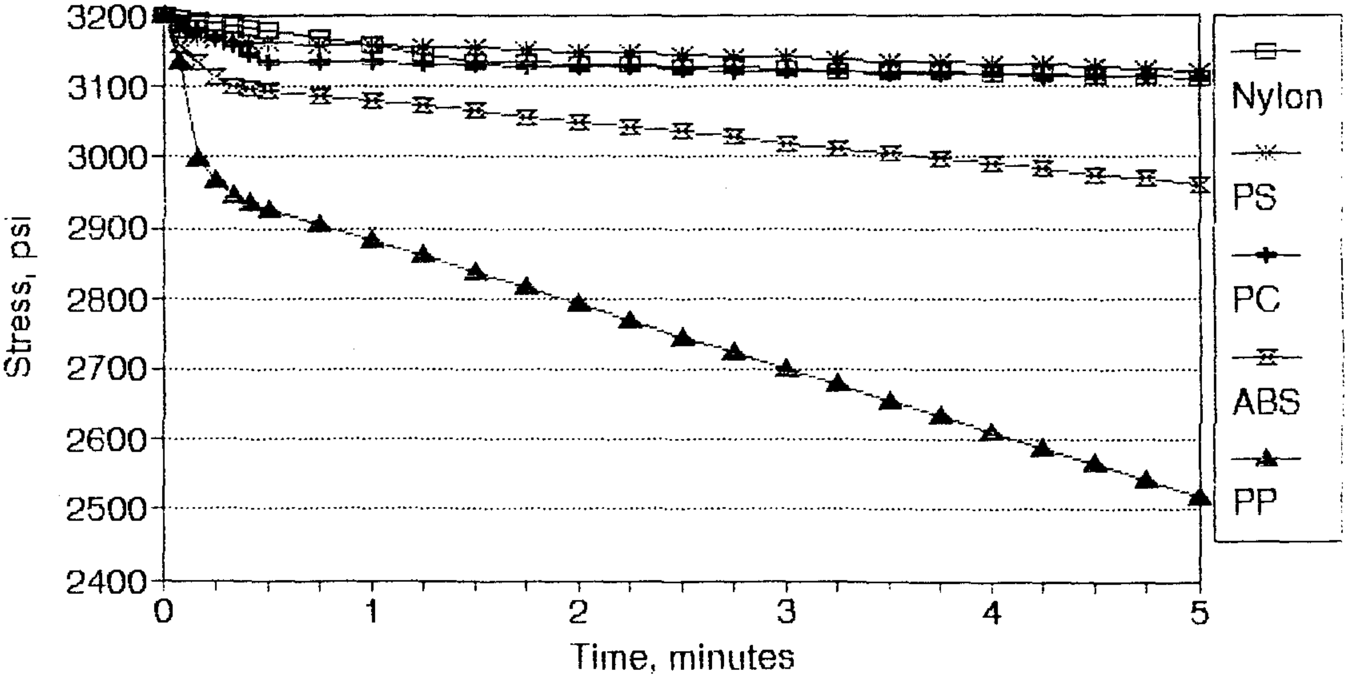


Fig. 2:

Results of sample relaxation experiment

5 plastics at same initial stress



CONCRETE REPAIR APPLICATIONS

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CONCRETE REPAIR APPLICATIONS
Using High Molecular Weight Methacrylate Monomers

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Key Words: High molecular weight methacrylate monomer, free radical polymerization, catalysts, concrete repair .

Prerequisite Knowledge: Basic organic chemistry and general chemical principles. Some understanding of polymers and polymerization reaction chemistry helpful but not necessary.

Objective: To provide a simple demonstration of how high molecular weight methacrylate monomers can be used to create a polymer composite that can be used successfully in concrete repair applications such as filling cracks in sidewalks and roadways, and especially in solving the pothole problem that plagues many municipalities during late winter and spring each year.

Materials and Equipment:

1. Sand (Pettinos No. 2 and 4)
2. Silica flour 120
3. Titanium dioxide (DuPont)
4. Cobalt acetyl acetonate
5. High molecular weight methacryalte monomer (Rohm & Haas)
6. Cumene hydroperoxide (Lucidol)
7. Make-shift wooden block and concrete forms
8. Disposable tongue applicators (for stirring)
9. Tissue wipes
10. Safety goggles and Platex-styled gloves.

Procedure:

Cumene hydroperoxide (73%) is thoroughly mixed with the high molecular weight methacrylate monomer selected. The freshly catalyzed monomer is quickly added to the dry mix of all the other components in a disposable polyethylene container of such size that it is about one-quarter filled. The resulting slurry is stirred until the dry mix is thoroughly wetted out with the catalyzed monomer. Test samples are then cast in miniature concrete molds or are filled directly into cracks in previously prepared concrete samples. Bulk cure is complete in 1-2 hours but surface cure takes 3-4 hours due to the inhibition of free radical polymerization reactions by oxygen in the air. For the purposes of a lecture demonstration, an accelerator can be added which effects cure of the bulk polymer in 5-10 minutes.

The actual mold that we have used to simulate a pothole consists of a $3X3X\frac{1}{2}$ inch Lucite frame mounted an a somewhat larger piece of concrete. The wetted mix is spooned into the frame with the disposable wooden applicator and "evened" off. It is then allowed to stand until cured. Since the surface will be uncured, a Handi-wipe can be used to dry the residual monomer before testing. Stress analysis and crack length can be used to evaluate the different monomers.

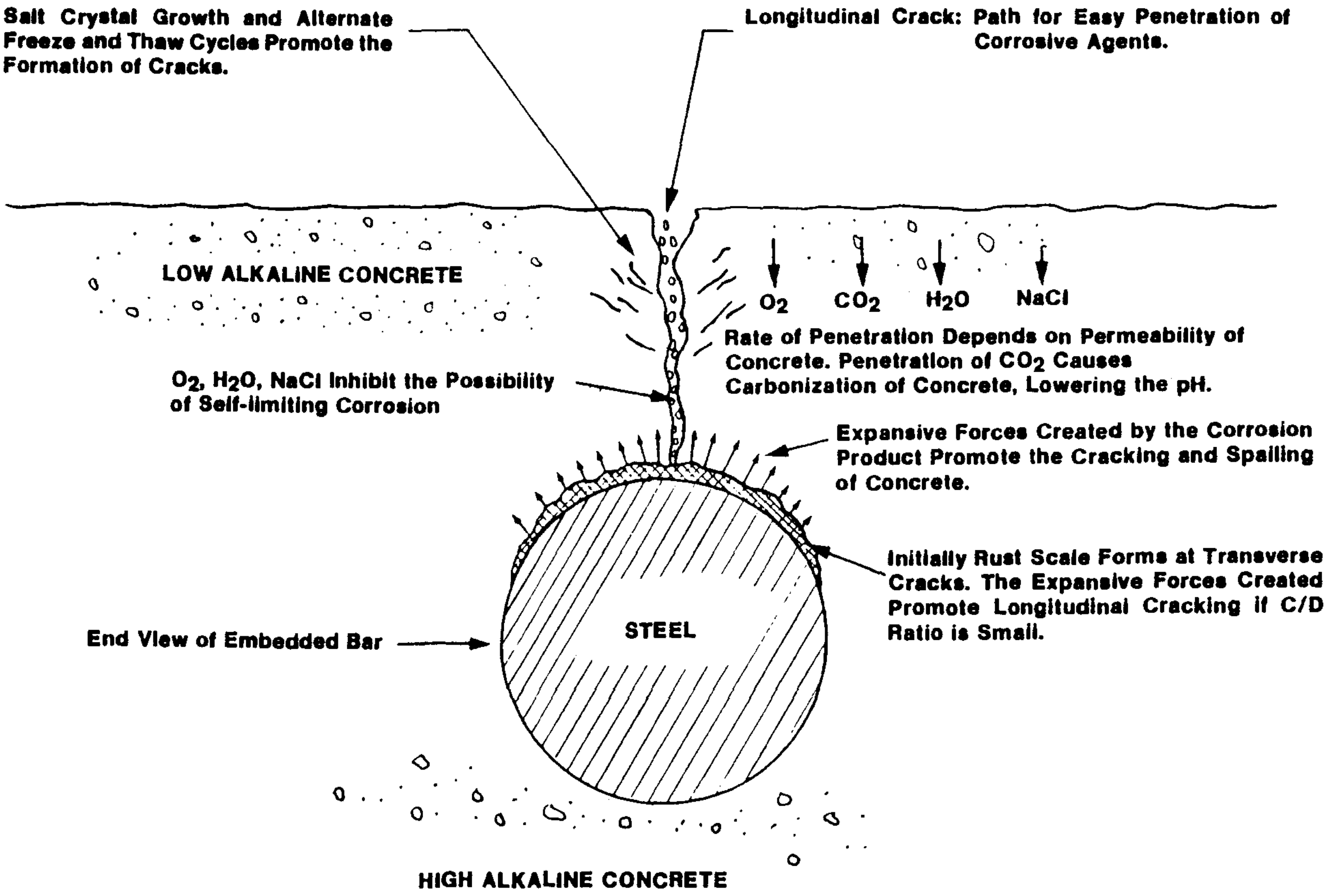
For our immediate purposes, the monomer that will be used is dicyclopentadienyl ethoxyethyl methacrylate. It is especially interesting because of the chemistry of its cure. Because of its low volatility, it is an excellent monomer for classroom demonstrations. It is easily synthesized.

Notes to the Instructor:

1. All of the above items are commercially available. General toxicity of all materials, including the monomers, is low. Suitable monomers can be purchased from several different sources or synthesized from easy-to-follow procedures available in the literature.

2. To visually demonstrate the strength of the material before an audience, use a hammer to violently stress the material on the side of the frame or directly on the surface of the cured polymer. Both tests will show the repair to adhere strongly to the old material, and in most demonstrations to be stronger than any original sample.

GENERAL MECHANISM FOR THE CORROSION OF REINFORCING STEEL IN CONCRETE.



STRENGTH/WEAKNESS COMPARISON OF CRACK REPAIR MATERIALS

PROPERTY	HMWM	EPOXY	MMA
SET TIME	S	N	S
BULK CURE	S	N	S
LOW TEMPERATURE CURE	S	N	S
VISCOSITY	S	W	S
TOXICITY	S	W	N
ADHESION	S	S	S
VOLATILITY	S	S	W
SUBSTRATE PREPARATION	S	W	W
FLAMMABILITY	S	S	W
ODOR	S	S	W

S=STRENGTH;W=WEAKNESS;N=NEUTRAL

TYPICAL MECHANICAL PROPERTIES OF HMWM POLYMER CONCRETE

	MONOMER 1540	MONOMER 1630	MONOMER 1680
COMPRESSIVE STRENGTH, PSI	9000	7200	5900
SHEARBOND ADHESION, PSI	735	900	650
TENSILE STRENGTH, PSI	1725	1100	800
FLEXURAL STRESS, PSI	2500	3000	1600
FLEXURAL STRAIN, IN/IN	0.002	0.013	0.043
FLEXURAL MODULUS, MM PSI	1.6	0.7	0.3

GLASS FRACTURE EXPERIMENT FOR FAILURE ANALYSIS

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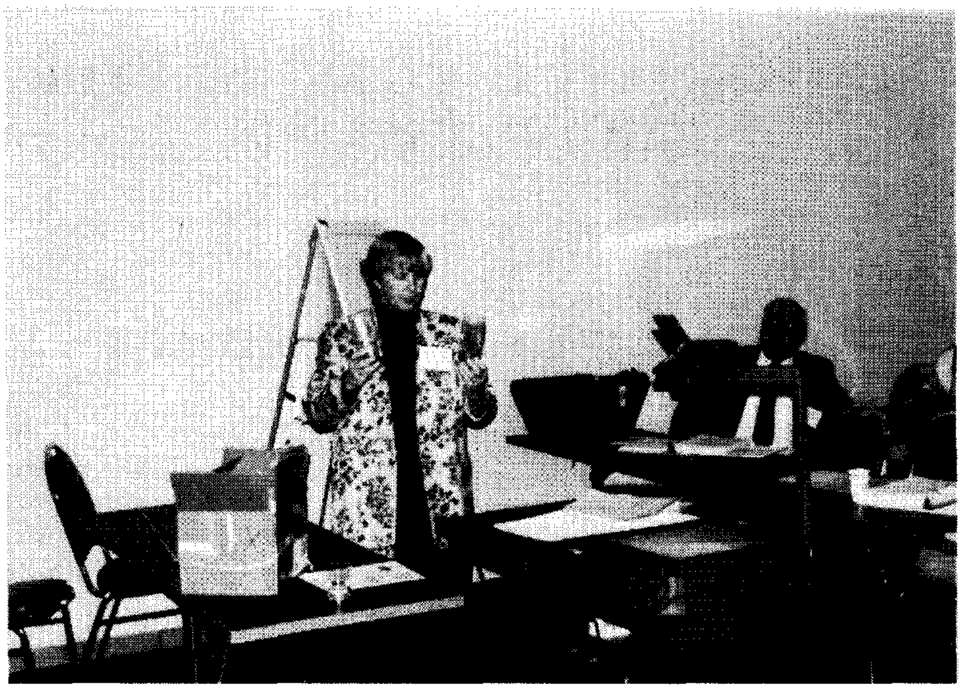

(Paper presented by Kathryn Forland)

GLASS FRACTURE EXPERIMENT FOR FAILURE ANALYSIS

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Key words: Fracture surface analysis, failure analysis, glass

Prerequisite knowledge: basic failure mode analysis and simple observation techniques

Objectives: 1)To provide the student with a learning experience in which they analyze a failure in a 2-4 hour period, and are able to verify their analysis with the people who have created the failure. 2) to learn to "read" a glass fracture and map the fracture path.

Equipment:

1. Drinking glasses with smooth sides approx. 60mm dia. by 150mm tall
2. 4-6 Ziploc quart size freezer bags
3. Safety glasses
4. Students dressed properly: long pants, shoes, shirts and long hair contained
5. Clear plastic tape
6. China markers or marking pens which will write on glass and not smear
7. Lab tongs
8. Several sheets of heavy brown wrapping paper.
9. 50mm wide masking tape
10. Hand held magnifiers 3X and 10X
11. Reference material pack (see bibliography)
12. Flashlight

Introduction:

The analysis of a failure involves proper procedure, careful study of the details and documentation of the facts. Often it is difficult for the student to fully appreciate the care and patience necessary in the examination and analysis of a failure. Secondly the student rarely sees an example in which he /she can do an actual analysis and get a definitive answer at the end of the exercise.

The idea of this exercise is to give the student practice in "reading fracture" surfaces and an appreciation for the care which is required to conduct a complete failure analysis.

Procedure:

This exercise requires the student to follow a procedure which will minimize the possibility for injury.

Safety glasses are to be worn during the entire exercise.

Each group of students 2-3 are given a drinking glass. This glass is placed in a plastic bag and sealed. Make sure to get all the air out of the bag. The entire parcel is then placed into a second plastic bag which is sealed after removing the air. The students are then instructed to break the glass. Students are allowed to break the glass any way they want to except high energy compression. For example, using an arbor press to crush the glass will tend to powder the glass and make it impossible to analyze. For this part of the exercise each group works out of site and ear shot of the other groups. This is necessary for security in divulging the method of breaking. **At no time during this period are the glasses removed from the plastic bags.** Should the bags be damaged during the exercise additional bags are provided to carry the contents back to the work tables.

The work area is covered with a sheet of heavy wrapping paper. The edges of this paper are folded to form an enclosure and taped to the table. The bags with the broken glasses are placed on the paper. When all the students have returned the glasses to the work area the groups are assigned to a glass they have not broken.

Safety :

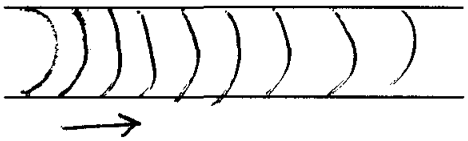
At this point the instructor demonstrates how to select and clean the glass fragments before beginning the analysis. The bags are opened and using lab tongs the larger pieces are removed from the plastic bag. Each piece is brushed with a piece of tape to remove the small particles of glass which would adhere to the surface. This procedure is similar to removing lint from a coat. Once the pieces are cleaned they are collected on the paper in an area away from the cleaning area.

When all the larger pieces have been removed from the bag and only the fines remain, the bag is sealed and the work area is cleaned using the same technique that was used to clean the glass pieces. The teacher should again remind the students of the safety requirements.

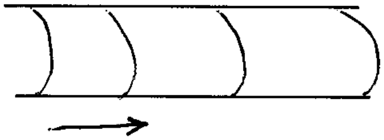
Analysis and reassembly.

The students are now allowed to proceed with the analysis using the reference material which they have been given. It works better if the reference material has been distributed to the students prior to the class so they can read it and become familiar with the fracture surface pictures. Students examine the individual fractured pieces, noting the nature of the fracture. From the references the student can "read " the fracture surface and determine those sections which are areas of fast and slow fracture. In addition one can determine the direction of the break by reassembling the glass with clear tape. The following illustrations are typical of some of the features which are definable in the surface of the fractured edge.

Crack propagation

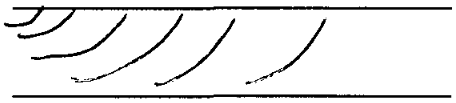


high speed/energy



low speed /energy

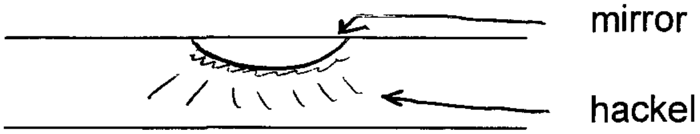
Direction



Bending at edge

bending at center

Fracture origin



The reassembling of the parts of a fracture is a violation of the principles of failure analysis. This breach of procedure, explained in the context of a learning experience usually satisfies the harshest student critic. In the reassembly, each piece is examined by eye and or low power magnification. The pieces are handled with lab tongs and only when absolutely necessary are they manipulated by hand. Each side of the piece is marked with an arrow indicating the direction of the fracture path and a letter f or s for fast or slow fracture mode. This process is carried out on all the pieces for each fracture surface.

When this procedure is completed the reassembly is begun. This part of the process takes about 1-1.5 hr.

Upon completing the assembly or that portion which can be completed, the student is asked to determine the mode of failure and make an oral presentation to the class. After the analysis group finishes its presentation the breaking group presents how they actually broke the glass. A class discussion follows on how the method and analysis differ.

Data:

The photo in Fig.1 illustrates the final product of the reassembly. It can be seen that the markings indicate not only the direction but the speed of the crack propagation.

Instructor notes:

This experiment is fun for both the students and the instructor. It teaches patience and the necessity for careful procedure and cooperation among the group members. I use this experiment in my Failure Analysis class which is a Sr./grad (573) class. The students are mature and understand the safety precautions necessary for the success of this kind of exercise. These students must still be cautioned about the potential hazards throughout the exercise.

Comments would be appreciated cvwhite@beretta.gmi.edu

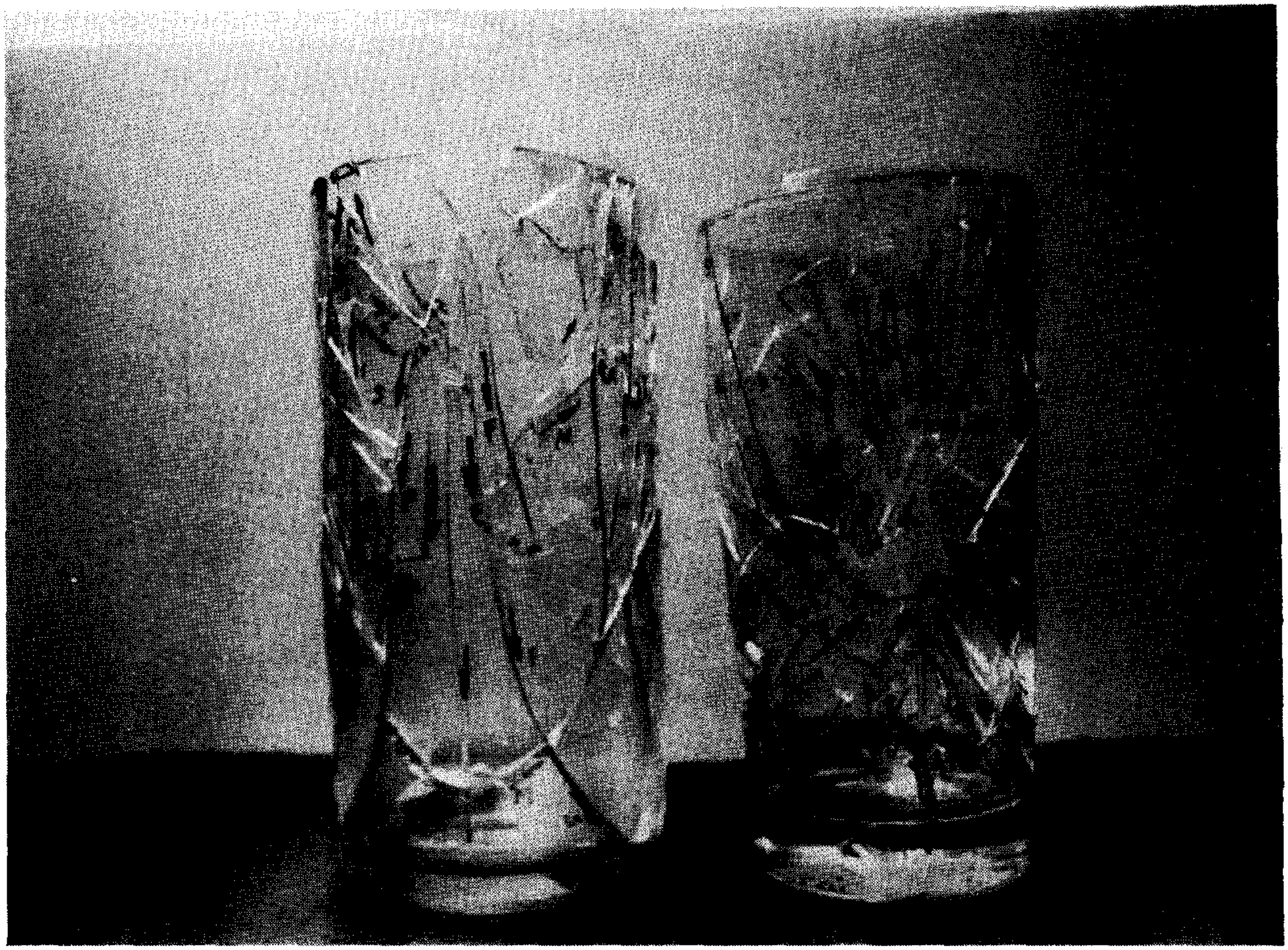


Figure 1.

References:

1. Stratton, D. R., CPP,: Reading the Clues in Fractured Glass. Security management, Jan. 1994, p. 56.

2. Preston, F. W.: Bottle Breakage - Causes and Types of Fractures, The Study Course on Mechanical Properites of Glassware with Particular Reference to Glass Containers. The Bulletin of the American Ceramic Society, February 1939, p. 35.

3. Orr, L.: Practical Analysis of Fractures in Glass Windows. Materials Research and Standards, January 1972, p. 21.

4. Preston, F. W.: The Propagation of Fissures in Glass and Other Bodies With Special Reference to the Split-Wave Front. Presented at the Annual Meeting, American Ceramic Society, Cleveland, Ohio, February 1931, p. 419.

5. Murgatroyd, J.B., B.A., F.S.G.T.: The Significance of Surface Marks on Fractured Glass. Presented at the Sheffield Metting, Journal of the Society of Glass Technology, June 17th, 1942, p. 155.

6. French, M. A.: How to Analyze Tempered Glass Fracture. Glass Digest, September 15, 1980, p. 50.

7. Nichols, R. T.: How to find the Cause of a Windshield Crack. Glass Digest, March 15, 1989, p. 56.

IMPACT OF FLAWS

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IMPACT OF FLAWS ON STRENGTH

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Key Words: Impact of flaws, laminated object manufacturing, structural ceramics

Prerequisite Knowledge: Knowledge of basic structural ceramic concepts is helpful but not necessary.

Introduction and Objectives

The purpose of this laboratory experiment is to demonstrate the impact of flaw size and geometry using the table-top test unit illustrated in Figure 1. Using this kit, a professor can dramatically illustrate the impact of flaws on load-at-failure for specific materials.

Ideally, high-technology materials should be flaw free. Realistically, flaws will always be present. These flaws should have a significant impact on the strength of a material and its suitability for particular production processes.

The material used in this test-apparatus is a green (un-fired) ceramic sheet. These sheets are flexible and are prepared from alumina ceramic powder and polymers. During the experiment students will test sheets that have dimensions of 3.8 x 7.6 cm. Three sets of sheets will be tested. Two sets contain different size flaws that have been cut into the tape using a laser. The third set of specimens does not contain a controlled flaw.

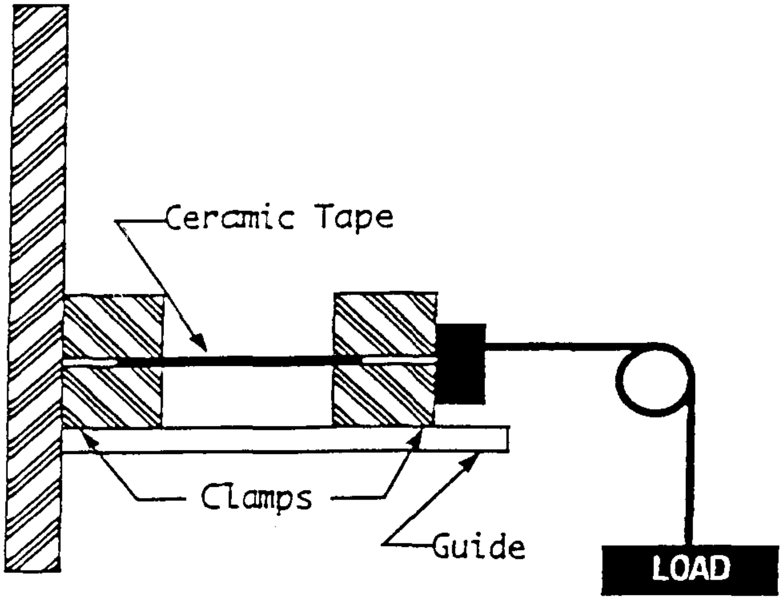


Figure 1. Test Unit

The test sheets used in this experiment were made by tape-casting, a specialized ceramic fabrication technique. Tape-casting is used to form sheets that have large surface areas with very thin cross sections. These ceramic sheets are essentially two dimensional in nature and are used as the basic building blocks for many electronic substrates and packages. Green ceramic tape has also been used as the feed material for a rapid prototyping process called laminated object manufacturing (1). Lone Peak Engineering originally developed this test apparatus to rate the suitability of different ceramic tape compositions for use in an automated lamination system.

Equipment and Supplies

1. Table-top test unit (see figures for details)
2. Green ceramic tape
3. Access to a scale
4. Data sheets
5. Calculator or computerized spreadsheet

Procedure

The professor should review how the test apparatus works with the students. The test apparatus is detailed in Figure 2. Overall operation of the test unit is fairly simple.

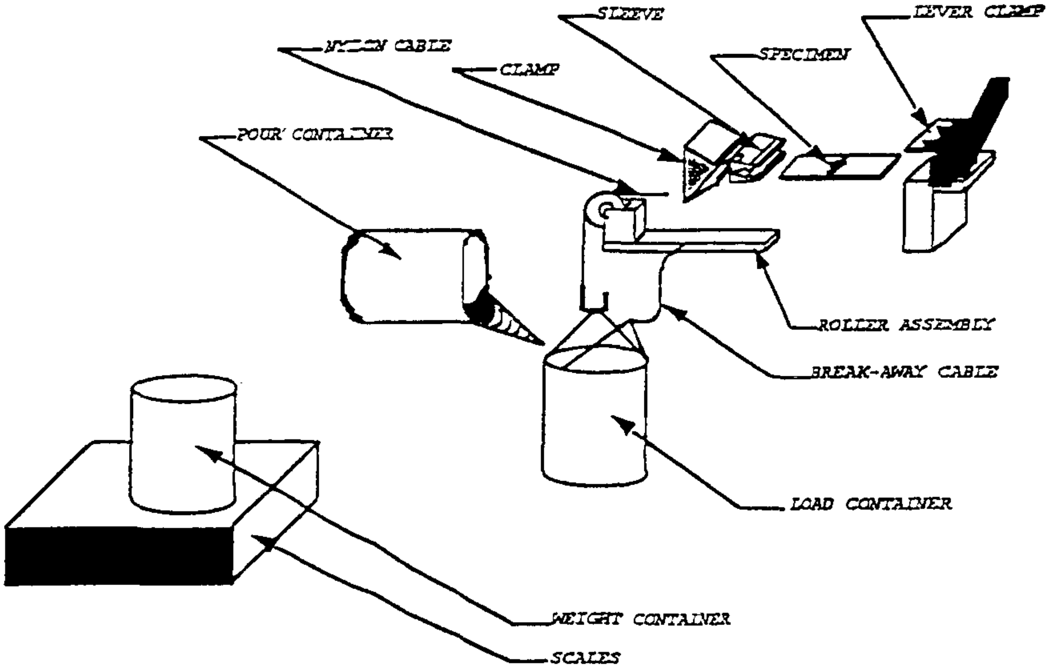


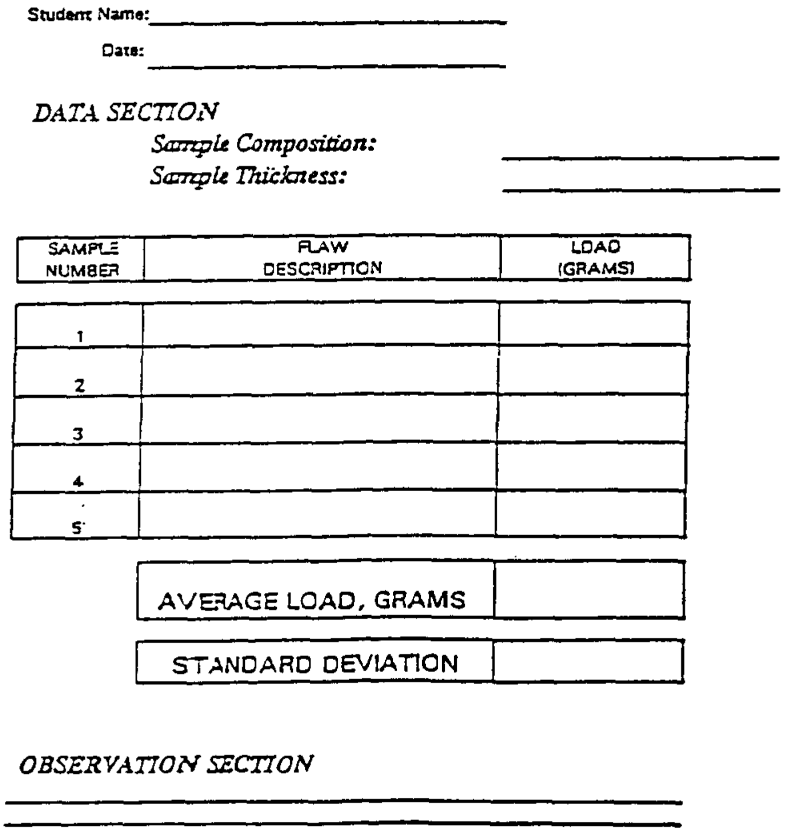
Figure 2. Detailed Test Unit

Steps

(1) Make tape measurements (subtract mylar backing thickness from total),
(2) Record information on data sheet,
(3) Remove tape from mylar backing,
(4) Insert tape into plastic sleeve,
(5) Place clamp over sleeve (clamp has nylon cable with hook attached),
(6) Attach empty load container to hook,
(7) Attach free end of tape to the lever clamp (clamp, nylon cable, hook, and load container should be attached to the other end of tape specimen before attaching to lever clamp),
(8) Tare weight container,
(9) Pour weights into load container at a steady rate until tape fails,
(10) After failure, pour weights from load container into weight container, and
(11) Weigh and record data.

Data Sheet

The student should use a separate data sheet for each specimen set. A sample data sheet is presented as Figure 3. It is recommended that students work in pairs and that 5 samples from each set be tested.



Student Name: ______________________

Date: ______________________

DATA SECTION

Sample Composition: ______________

Sample Thickness: ______________

SAMPLE NUMBER	FLAW DESCRIPTION	LOAD (GRAMS)
1		
2		
3		
4		
5		

AVERAGE LOAD, GRAMS	
STANDARD DEVIATION	

OBSERVATION SECTION

____

____

Figure 3. Sample Data Sheet

Have the students calculate the standard deviation for each set of sheets using the formula below.

$$s = \sqrt{\sum_{j=1}^{(N-1)} (X_j-F)^2 /(N-1)}$$

Where:

N = the total number of specimens measured

X_j = the failure load of each individual specimen

F = the average failure load

Test Specimen Details

The test specimens have the following dimension.

(1) Width = 3.8 cm
(2) Length = 7.6 cm
(3) Flaw length (2a) where (a) is either 0, 0.48, or 0.95 cm

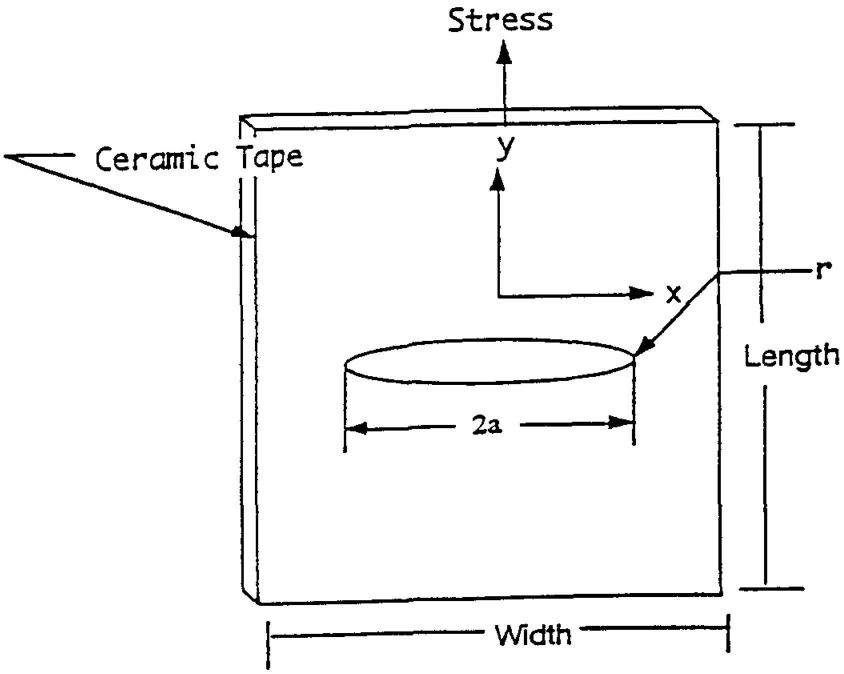


Figure 4. Ceramic Test Specimen

Instructor's Notes

The instructor should have the student's examine the failure area. Perhaps a drawing, such as presented below, would be helpful. The specimen in Figure 5 broke uniformly. The students should write up a report to go with the data sheet that summarizes their findings. It would be helpful if the instructor covered some design theory as it relates to flaws in materials before the students begin the experiment.

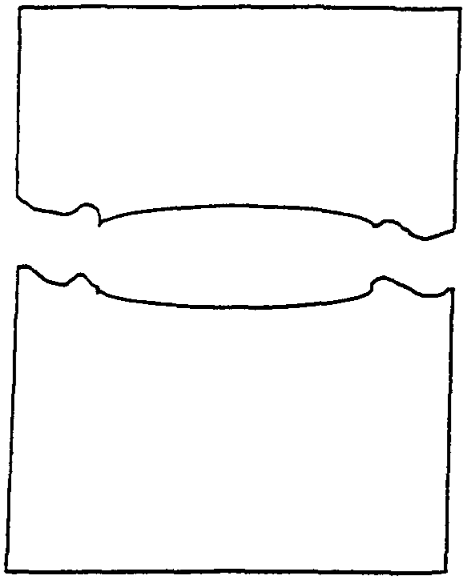


Figure 5. Failure of a Flawed Test Specimen.

References

1. Griffin, C.W., Daufenbach, J., and Feygin, M., flexible Manufacturing of Advanced Structural Ceramics Using Laminated Object Manufacturing Techniques, Phase I DARPA, Arlington, Virginia under Contract DAAHO1-92-C-R244, DAAHO1-93-C-R350, 1993.

Source of Supplies

It would be possible to make your own table-top tester using the details provided by the different drawings. Green ceramic tape can be purchased from commercial vendors or prepared by students in a University laboratory setting. However, LPE has prepared this experiment as a complete kit. The kit includes:

1. Table-top tester
2. Supply of ceramic tape, with and without controlled flaws.
3. Data sheets

Please contact Lone Peak for additional information.

STUDYING MACROSCOPIC YIELDING IN WELDED ALUMINUM JOINTS USING PHOTOSTRESS

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Key Words: Photostress, Photoelastic Coatings, Stress Analysis, Material Characterization, and Mechanical Behavior.

Prerequisite Knowledge: Senior in College having a basic understanding of mechanics of materials and mechanical behavior of materials. The instructor should give the student a basic lecture in the theory of Photoelasticity and its application to strain measurement.

Objective: To observe the macroscopic yielding characteristics of a welded aluminum joint using photoelastic coatings. To determine the variation in the stress-strain behavior on a point by point basis in the weld metal and the heat affected zone.

Equipment:

1. GTAW aluminum plate (2219-T87 base plate and 2319 filler)(or appropriate substitute).
2. Rubbing alcohol (or acetone).
3. Coarse and fine silicon-carbide paper (220 grit and 400 grit).
4. Gauze sponges.
5. Q-tips.
6. Flat wooden mixers.
7. Metal surface cleaner (M-prep conditioner A).
8. Neutralizer (M-prep neutralizer 5A).

9. Photoelastic Coating (PS-1).

10. Adhesive (PC-8).

11. Reflection polariscope with a uniform field compensator, tele-microscope, and a digital strain recorder.

12. Universal Testing Machine.

13. 1000 speed color film.

14. Automatic camera.

15. Curve fitting program on a computer.

Introduction: Many structures are fabricated by welding standard structural members such as beams, columns, angles, etc. The process of welding involves melting, phase change, and intimate inter-granular mixing of the two metals to be joined. Heating and cooling during welding is known to alter the mechanical properties of the weld metal and the metal surrounding the weld, called the heat affected zone (HAZ).

Characterization of yielding behavior in weld material and the surrounding HAZ is very important for a complete understanding of the response of welded joints to loads. Knowledge of the mechanical properties and the macroscopic yielding characteristics is very important for accurate modeling and design of structures having welded joints.

There are several experimental methods which can be used to determine the distribution of strain in structures and other machine components. The most common method is the use of electrical resistance strain gages. Unfortunately, strain gages provide data only at points where they are mounted on the structure and little is learned of the yielding characteristics of other regions. The method of Photostress, also known as the method of photoelastic coatings, is a full field optical technique which allows the study of macroscopic yield behavior in the area of interest. At the same time, it allows for measurement of strain on a point by point basis. A detailed description of the theory of photoelastic coatings can be found in [1].

The Photostress technique allows for measurement of maximum in-plane shearing strain using the equation:

$$\gamma = \epsilon_1 - \epsilon_2 = Nf_\epsilon$$

where $f_\epsilon - \lambda/2TK$ and

γ is the maximum in-plane shearing strain,

ϵ_1 and ϵ_2 are the principal strains,

N is the fringe order,

f_ϵ is the fringe value,

λ is the wavelength of light = 22.7×10^{-6} in,

K is the calibration constant of the coating, and

T is the thickness of the coating used (0.01 or 0.02 in.).

Procedure:

Specimen Preparation: Machine a dog bone specimen (Figure 1) from welded aluminum plate based upon the capacity of the testing machine and the ultimate strength of the material. Insure that the centerline of the weld is at the center of the specimen. Using the coarse silicon-carbide paper, abrade the area of interest which is the weld metal and the area surrounding the weld metal (approximately 2 inches on either side of the weld centerline). Clean the abraded area with rubbing alcohol (or acetone) and gauze sponges to remove foreign substances. Put a few drops of metal surface cleaner (diluted acid) on the surface and wet-lap it with fine silicon-carbide paper. Clean the surface with a gauze sponge, add a sufficient quantity of neutralizer (diluted basic solution) and, using a Q-tip, clean the surface thoroughly. Before the surface dries, wipe it clean with a gauze sponge.

Cut the photoelastic coating (PS-1) to the required dimensions (see Figure 1) using sharp scissors. Care should be taken not to snap the scissors as this results in damage to the coating material. Using a flat wooden mixer, spread the adhesive (prepared by your instructor) evenly on the surface of the specimen. Place the cut photoelastic coating sheet along one edge of the specimen and slowly press it into position while working out the air bubbles in the process (see Photo 1). After approximately 45 minutes, make a fillet of the adhesive along the edges and remove the excess adhesive. Let the specimen cure at room temperature for 48 hours. Then take the specimen and, using a permanent ink marker (Figure 1), mark the locations where you want to measure strain. Now the specimen is ready for testing.

Testing:

Based on the capacity of the testing machine, prepare a loading sequence to load the specimen in a reasonable number of steps to about 90% of the ultimate strength of the weld metal. Using Photograph 2 as a reference, set up the experiment. Program the universal testing machine to load in the predetermined loading sequence. Have the camera loaded and focused to photograph the fringe patterns. After the experiment set up has been completed, turn down the lights in the lab. Adjust the direction of the polariscope light source to see that there is no reflection off of the coating material and that the light shines uniformly on the photoelastic coating. Start loading the test specimen and, at each load step, take a photograph of the fringe pattern and, using the telemicroscope attached to the reflection polariscope, measure the strain as explained by your instructor in the introductory lecture. Tabulate load and strain (micro strain) measured at each point as shown in the sample below:

Measured Shearing Strain

Load (lbs)	**Centerline**	**Quarter-point**	**Half-point**	**Three-quarter point**	**One-inch point**
2000	800	600	450	200	190
4000	1100	880	630	280	250

Photograph 2 shows the progressive development of fringe patterns for a 1.4" thick, 2219-T87 welded aluminum joint and the corresponding stress values. It can be clearly seen that the fringe development gives a good representation of the yielding characteristics of the weld metal and the surrounding heat affected zone. This understanding is critical in developing analytical and finite element models for welded joints.

After measuring the strain, convert the load values into the corresponding stress values based upon the cross-sectional area of the specimen and, using a curve fitting program, plot stress-strain curves for each of the points. Figure 2 shows stress-strain curves for the 1.4" thick welded aluminum joint shown in Photograph 2. The stress-strain curves show the highly non-linear behavior of the welded joint and the variation in mechanical properties from the center of the weld outward to points one inch away. This technique can be used not only on flat surfaces but can also be used on curved surfaces if the photoelastic coating is contoured to the shape of the surface while it is in a semi-polymerized state.

Notes to Instructor:

All materials and techniques required for using Photostress can be obtained from Measurements Group, P. O. Box 27777, Raleigh, North Carolina 27611. Telephone (919)365-3800. They also can provide a wide variety of literature on using photoelastic coatings. A few of their bulletins are listed in the reference section. They will be particularly useful in conducting the experiment described.

References:

1. Coker, E. G., and Filon, L. N., 1931, "A Treatise on Photoelasticity," Cambridge University Press.
2. "How to Select Photoelastic Coatings," Measurements Group, Inc., Literature, Raleigh, NC, TN-704, (1978).
3. "Photostress Instrumentation," Measurements Group, Inc., Literature, Raleigh, NC, Bulletin 315-B, (1989).
4. "Automatic Data Acquisition," ADA-1 through ADA-8, Measurements Group, Inc., Literature, Raleigh, NC.
5. "Operating Instructions and Technical Manual-Strain Measurement with 030-Series Reflection Polariscope," Measurements Group, Inc., Literature, Raleigh, NC.
6. "Instructions for Bonding Flat and Contoured Photoelastic Sheets to Test-Part Surfaces," Measurements Group, Inc., Literature, Raleigh, NC, IB-223-F, (1982).
7. "Instructions for Using PC-8 Adhesive," Measurements Group, Inc., Literature, Raleigh, NC, IB-231, (1982).

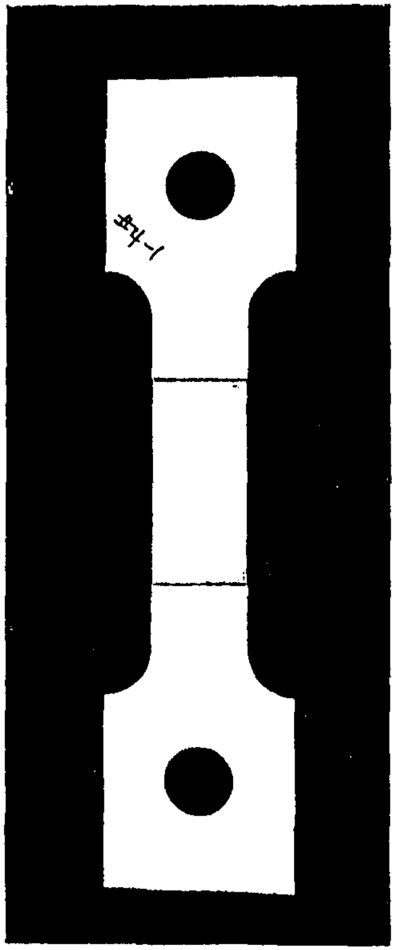


Photo 1. Typical Specimen

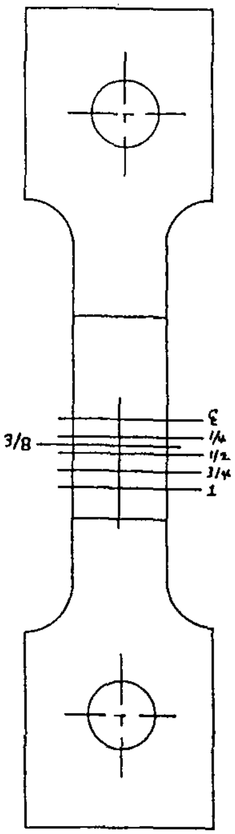
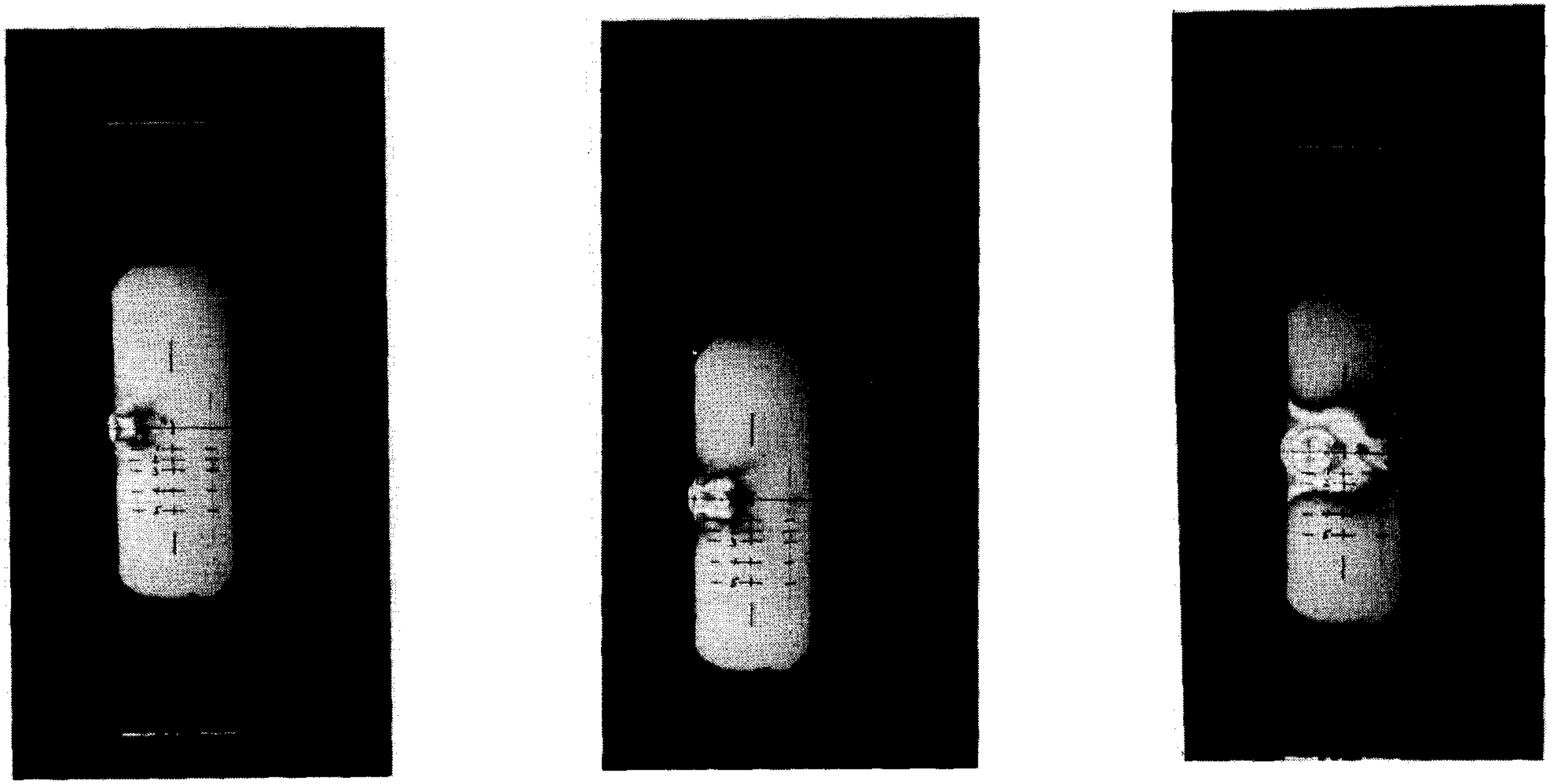


Figure 1. Sketch of Specimen



(a) 25,260 psi (b) 28,890 psi (c) 36,100 psi

Photograph 2. Shear Strain, Through Weld, Bead On

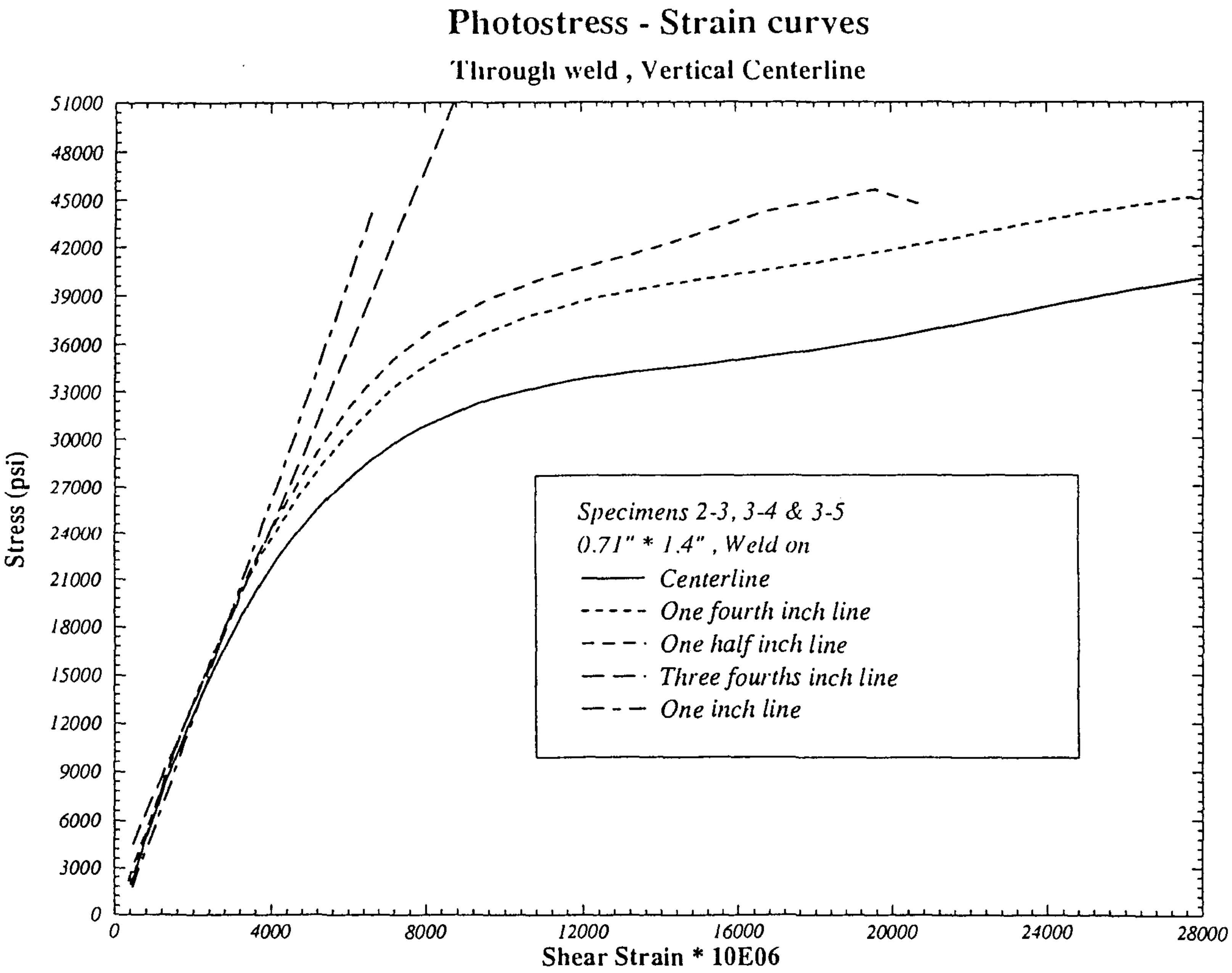


Figure 2. Behavior, Weld On, Vertical Centerline

BUBBLE RAFTS, CRYSTAL STRUCTURES, AND COMPUTER ANIMATION

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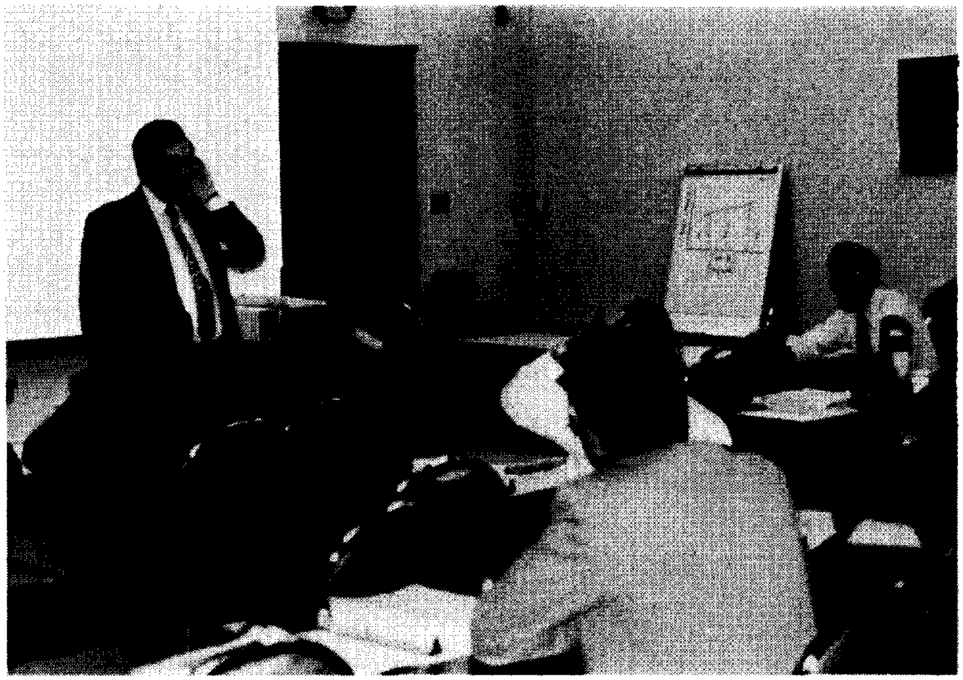

BUBBLE RAFTS, CRYSTAL STRUCTURES, AND COMPUTER ANIMATION

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KEY WORDS: Crystals, bubble rafts, computer animation.

PREREQUISITE KNOWLEDGE: The student should be familiar with the basics of materials science and chemistry. Levels at which these experiments are performed are second semester junior and either semester senior year. The students are first given lectures on crystal structures, solids, and basics of crystallography, defects, and diffraction. No microscopy is needed and only a computer and an overhead projector are really essential. A video filmed by the instructor showing the bubble formation and the computer animation gives the student an idea of what is expected.

OBJECTIVES: To illustrate the process atom-by-atom structure formation using bubble rafts and to show three-dimensional animated stick, ball, and hard-sphere animated computer models using public domain software on one 360K floppy. This should aid the sometimes futile attempt by both the professor to teach and the student to visualize crystal structures and defects.

EQUIPMENT AND SUPPLIES:

(1) Pyrex flat, square dishes (2 per student)
(2) Oleic acid, triethanolamine, glycerine, miscellaneous chemicals,
(3) A stirring rod,
(4) A pin,
(5) A 286/386/486 PC with DOS or Windows,
(6) Public domain software (provided).

INTRODUCTION: Using simple chemicals and materials and using simple, public domain software, the student can become better acquainted with crystal structures, defects, and stacking in two and three dimensions. Crystal symmetry and the density of atomic packing using the hard sphere model can be easily visualized. Rotation of the specimens in three dimensions and creation of varying types of defects can be easily accomplished in this one session laboratory.

Part I:Crystal Structures: Crystalline materials tend to have long-range order in the position and stacking sequence of atoms or molecules. On the other hand are amorphous solids, which do not show any long-range order and only have directional bonding between atoms. The simplest crystal structures are composed of pure elements and exhibit isotropic properties due to non-directional interatomic bonds. These atoms pack together in such a way as to minimize the open space in the structure and form what we call close-packed crystal structures. There are fourteen ways in which atoms can arrange themselves in space. These are called the **Bravais Lattices**, shown in Figure 1 below. Looking at these, one can

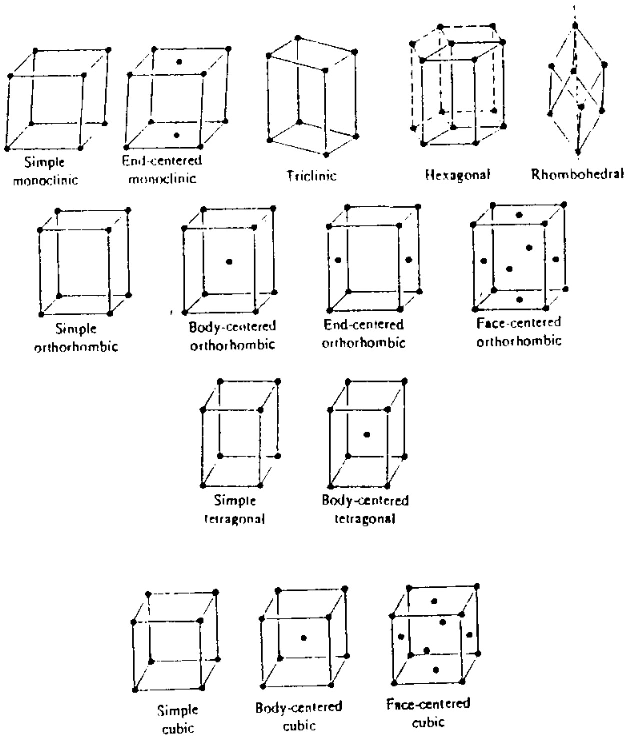


Figure 1
The fourteen Bravais Lattices

observe that they have different rotational symmetries, Miller indices of their planes, holes, and packing densities. By examining models of these crystal structures, one can become more familiar with their physical properties and bonding relationships. Numerous references give excellent introductions to crystallography[1,2,3].

Part II: Imperfections: In today's world, we know there is no such thing as a perfect crystal, so we can visualize a real crystal as the superposition of discrete imperfections onto a perfect crystalline lattice. These imperfections may be conveniently classified with respect to their dimensionality[2]. Zero dimensional imperfections are known as **point defects**. Point defects are located at defective regions of a crystal that are confined to a volume of a point (a sphere of atomic dimensions). Some examples of this class are vacant lattice sites and impurity or foreign atoms. Examples of these types of imperfections are shown in Figure 2.

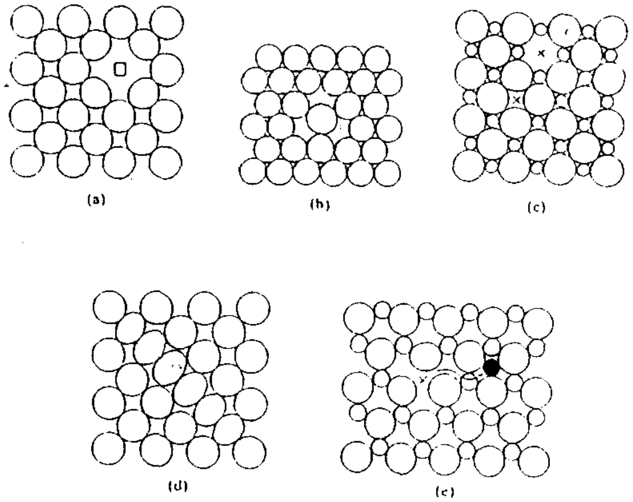


Figure 2
Classifications of point defects
(a) vacancy; (b) divacancy; (c) ion pair vacancy (Schottky defect); (d) interstitialcy;
and (e) displaced ion (Frenkel defect)

The one dimensional class of imperfections are known as **line imperfections**, or, generally called, **dislocations**. There are defective regions of the crystal that extend through the crystal along a line. This line is not necessarily straight, and it may be curved or even be a loop. **Interfaces** are two dimensional imperfections that serve as **boundaries** between regions of a crystal which have different ordering or arrangements. A depiction of a grain boundary is shown in Figure 3. An interface is the transition region in which the configurational arrangement of atoms of a crystal is interrupted. The three-dimensional class of defects are known as **bulk defects**. Bulk defects are macroscopic defects that represent an inhomogeneity on the shape or structure of a solid. Some typical bulk defects are voids and cracks. Two of these are shown in Figure 4.

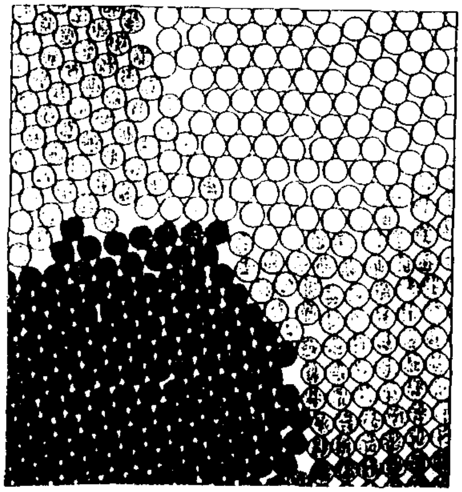
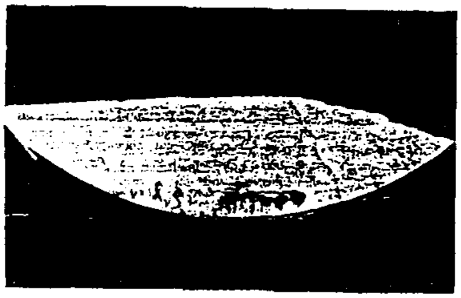


Figure 3
Grain boundary



shrinkage cavities in a steel casting.

inclusions in structural steel

Figure 4
Bulk defects

These classifications of dimensionality range in size from microscopic (10^{-14} m) to the macroscopic range of fractions of meters. A depiction of these is shown in Figure5

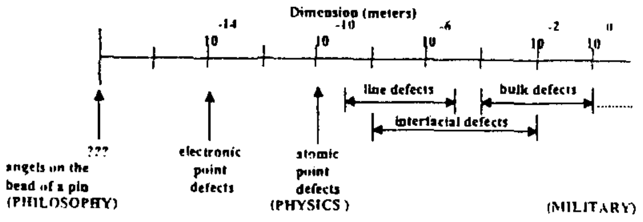


Figure 5
Size classification of defects in solids

In addition, some defects may be removed (or moved) by annealing, making larger regions of perfection in the crystal, as shown in Figure 6.

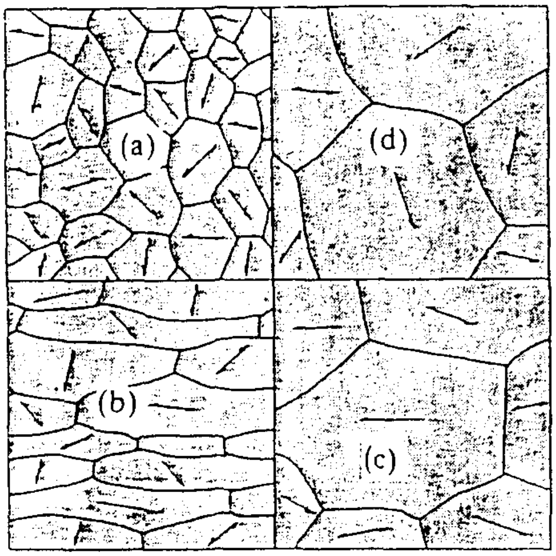


Figure 6
Annealing to enlarge regions of crystallinity
(depictions (a) through (d) showing annealing vs time)

THE EXPERIMENTS:

Computer generated crystal structures:

In order to provide the student with another tool for visualizing the three dimensional structures in space, a computer visualization program called "Chemview" was employed. It is a public domain, DOS based program costing $3.50 from Sector Systems Company, Inc., Marblehead, MA. One can construct as complex an organic, polymeric, or crystalline structure as time permits. This figure can then be rotated in 3-D space and viewed at any angle. If one wishes to incorporate this construct into a report, a screen grab may be used or the view may be printed directly form the program. One such construct, a dodecahedron, is shown (as grabbed from the screen) in Figure 7. Usually, the student will take the copy of the program from the laboratory and spend hours making cutaways of planes in crystal structures as well as challenging variations of diamond cubic for binary, ternary, or higher compounds. The instructor is free to use the program as he or she sees fit, in accord with classroom or other laboratory exercises. Other more expensive programs[6] may be substituted for this if funding permits. Motivationally, this departure from, 2-D drawings and Styrofoam models is superior for retention and creativity.

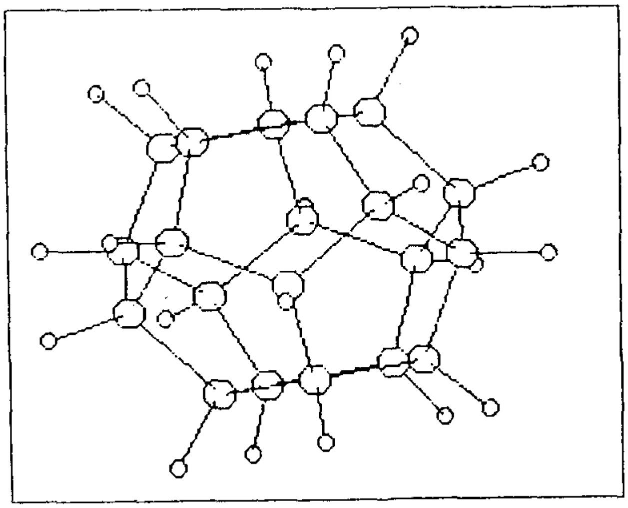


Figure 7
Computer generated dodecahedron structure rotated about an axis

The bubble raft: The bubble raft is an experiment originally designed and built by Bragg and Nye[4] in 1947 to overcome the student's apparent inability to visualize defect structures in hard-sphere model solids. In this experiment, crystal structure of a metal is represented by an assemblage of millimeter-sized bubbles, floating on the surface of a soap solution. The bubbles are blown beneath the surface of the solution under constant air pressure, creating a highly uniform array of bubbles (atoms) held together by capillary attraction, simulating quasi-free electron forces. This experiment shows representations of structures in two and three dimensions, such as point defects, grain boundaries[5], faults, slip, recrystallization, annealing, and strain, all at room temperature. These structures can be easily observed on an overhead projector.

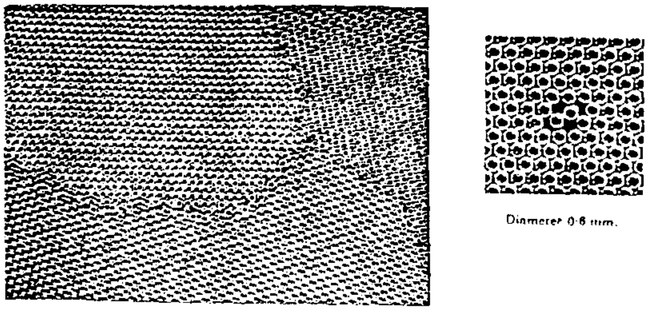
Procedure

1) To 15.2 cc of oleic acid,
2) Add 50 cc distilled H_2O.
3) Mix (shake well) together.
4) Mix with 73 cc of 10% solution of triethanolamine and the mixture made up to 200 cc.
5) Add 164 cc pure glycerine.
6) Let stand and draw of clear liquid from below.
7) This solution can be diluted in three times its volume of water to reduce its viscosity.
8) From this decant, yield is approximately 300 mL of usable mix.
9) Dilute with 150 mL of H_2O to make soap solution for trays.
10)To make solution: 1:4 bubble solution to water.

Using an eyedropper as a nozzle held at least 0.5 cm below the surface of the solution, blow the bubbles using an aspirator bottle, making sure that the bubbles form at a slow and uniform rate. Bubbles should be of the order of 1-3 mm in diameter. Using an aspirator bottle held at a given level, a uniform pressure can be delivered to the bubble nozzle orifice. In the same tray, these bubbles may be used to simulate grain boundaries, dislocations, vacancies, slip, recrystalization, and annealing. By introducing smaller or larger bubbles, impurity atoms may be simulated. By putting a piece of flat wood into one side of the solution, simulation of stress can be achieved. The meeting of two dislocations results in an observed vacancy formation as well. Typical examples of the bubble simulations are shown in Figures 8(a) and 8(b).

Oblique view of three-dimensional raft.

Figure 8(a)



Grain boundaries

Diameter 0·6 mm.

Figure 8(b)

Observations of defect structure using bubble rafts

Reporting: On the photographs taken or on a suitable drawing, indicate the positions of the following: (a) a vacancy; (b) a grain boundary; (c) a triple point; (d) a dislocation; (e) an impurity atom. Following your observations of the bubble raft and of your studies of imperfections, you should be able to simulate the following:

1) A polycrystalline structure and crystal or grain growth, grain boundaries. How did you do it? What happened?

2) Vacancies and impurities. How did you do it? What happened?

3) Dislocations showing their motion (apply a stress to the raft), their interaction, mutual annihilation, and climb of dislocations. Please indicate graphically and narratively what happens.

Using the computer modeling programs, construct a structure of your choosing. This should be of at least the difficulty level of a face centered cubic structure.

Alternate scenarios of diffusion (using dyes), climh (propagating a defect), and putting large and small bubbles on opposite sides of the tray (solid solution formation) are possible with some practice. Once again, making the bubble material available to the student, even after the laboratory sessions, allows for a safe and realistic simulation of defects.

Summary: Using simple chemicals and materials, students can familiarize themselves with the basics of crystal structures, defects, and three-dimensional computer models. A full experiment is provided for implementation of this laboratory. By employing inexpensive, public domain, computer programs, meaningful visualization and retention of three dimensional structures are possible.

These experiments were developed by seniors in the process of their senior design project capstone courses at Western New England College.

REFERENCES:

1. Van Vlack, L. H., **Elements of Materials Science and Engineering**, 6th Ed., Addison-Wesley Publishing Company, Reading, MA 1989.
2. Jacobs, J. A. and Kilduff, T. F., **Engineering Materials Technology**, 2nd Ed., Prentice Hall Publishing Co., West Nyack, NY 1994.
3. Shackelford, J. and Alexander, W., **The CRC Materials Science and Engineering Handbook**, CRC Press, Boca Raton, FL 1991.
4. Bragg, L. and Nye, J. F., **A dynamical model of a crystal structure**, Proc. of the Royal Soc. of London, A190, 10. 1947.
5. Ishida, Y., **Bubble raft grain boundaries**, in Order Structures, Chapman Hall, 1972. pp.75-82.
6. Chemintosh, Autodesk Modules, Chemwindows, etc.

SOURCES OF SUPPLY: The laboratory supplies and chemicals were obtained from Cole-Parmer and Fisher Scientific. All other supplies are easily obtained from local university supply houses. The software is public domain and is available from Sector Systems Software, Marblehead, MA.

IN-CLASS EXPERIMENTS: PIANO WIRE AND POLYMERS

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In-Class Experiments: Piano Wire and Polymers

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Overview

A number of challenges arise when teaching an introductory materials engineering course. Many students lack hands-on experience with hardware, especially with the variety of currently available materials. Laboratory experience is an effective way to deal with this challenge, but the available time, money, and facilities often constrains the amount of laboratory exposure. One solution that allows students to explore material response in less time and a lower cost is to have small in-class experiments.

In the introductory materials course at Rose-Hulman Institute of Technology, a number of small experiments have been used to get the students actively involved with materials in the classroom. These are team-based experiments in which the students explore material response and attempt to relate material behavior to material processing and microstructure.

Two short, inexpensive in-class experiments are described in this paper. In Part One students experiment with music wire to reinforce their understanding of processing, microstructure and properties of steel. In Part Two, some readily available polymer parts and toys are used to discuss viscoelasticity, and polymer microstructure.

Key Words:

Part 1: Cold Work, Heat Treatment, Steel
Part 2: Polymers, Viscoelasticity,

Prerequisite Knowledge:

Part 1: Some knowledge of the processing-microstructure-property relationship for steel.
Part 2: Difference between thermosets and thermoplastics, microstructure of polymers, deformation mechanisms of polymers.

Objective:

1) to help the students develop an intuitive understanding of materials by hands-on exposure to material behavior and correlation with processing and microstructure.
2) to continue to encourage cooperative learning[1] through the use of a team-based problem solution. The goals for the individual experiments are listed in the class handouts.

Part One: Music Wire

The following are abbreviated versions of the course handouts used for the music wire experiment. To encourage teamwork, one handout is given per group.

Properties of Music Wire

Task: To qualitatively examine the mechanical properties of three pieces of music wire and deduce the processing that produced those mechanical properties. Justify your deductions from a microstructural perspective using the information and tools learned in class.

Materials: Three 10-15 cm long samples of 20 Gauge (0.045 in diam.) steel music wire. Each sample has been processed differently.

- Specimen A - Shiny.
- Specimen B - Not shiny, blue dye on one end.
- Specimen C - Not shiny, no dye at either end.

Group member roles: For this work identify a recorder and a runner for your team. Their responsibilities are:

Recorder - Record all group observations on the supplied sheet.
Runner - Act when needed according to the following instructions.

Instructions:

1. Have runner acquire one of each type of specimen for the group from the table at the front of the room.
2. Each person in the group will try to bend each specimen
3. As a group, discuss the mechanical behavior for each specimen and record your description.
4. As a group, deduce and **justify**:
   - What type of microstructure each specimen has
   - What type of processing each specimen has seen.

Background:

Music wire is a high purity plain carbon steel with carbon content of approximately 0.8%. It is sold in the cold worked condition (formed by wire drawing). The strength in the cold worked condition is typically between 200 and 400 ksi. As purchased, it should be able to bend back on itself (180° bend) without fracture. The primary industrial application of music wire is for springs.

Properties of Music Wire
Recorder Sheet

3. Description of the mechanical properties (qualitative) of the specimens.
   - Specimen A
   - Specimen B
   - Specimen C

4.

Specimen	Microstructure	Justification
A		
B		
C		

Specimen	Processing	Justification
A		
B		
C		

Instructor Notes: In the piano wire experiment, each team of three to four students receives a packet of three color-coded short lengths of piano wire that have undergone different thermo-mechanical treatments. The students are instructed to bend each of the wires, noting the relative force required for each and comparing the materials with respect to strength and ductility. After testing, each student team must answer a series of questions that require them to deduce the processing and consequent microstructure of the steel. All deductions must be accompanied by reasoned justification.

- Specimen A was the music wire in the as-received conditioned - cold worked to high strength.
- Specimen B was heated to 1600°F for one hour and quenched in water.
- Specimen C was heated to 1600°F for one hour and furnace cooled.
- As an alternative, a specimen D that has been austenitized, quenched, and tempered could be included, but tends to create significant confusion.

At the time of the experiment, the students had received a thorough grounding through lecture in cold work and annealing, and had an introduction to steel microstructure and heat treating. Consequently, the student's could be expected to recognize that the very brittle response of Specimen B was due to a martensitic microstructure caused by being austenitized and quenched. Likewise, the shiny appearance of Specimen A indicated no thermal processing and its strength and ductility was good evidence that the material is still in the as-received condition. The very soft and ductile Specimen C presented the students with the most difficulty. Ninety percent of the twenty groups correctly identified the microstructures of Specimen A as pearlite and Specimen B as martensite. For Specimen C, one third of the teams correctly deduced pearlite. Of the others, one third deduced bainite and one third other responses.

The small group discussion in this exercise allows two important things to take place. First, the students must convince each other that their answer is correct. This encourages them to apply reasoning rather than memorization to solve the problem. Second, discussion provides an opportunity for the students to sort out the meanings of the new vocabulary. Past experience indicated that many students were confused by the quantity of new vocabulary describing the microstructure and heat treatment of steel.

The results of the lab indicated several things. The fact that many of the students expressed surprise at the differences in mechanical behavior showed the lack of experience with materials. Observation of the teams showed that the cooperative effort of the group was helpful in reaching the correct responses. The groups making the mistakes were often guessing and were not able to relate the book values for hardness of the microstructures to the relative strengths of the wires.

This experiment could be done at the outset of introduction to steels as well, but less could be expected of the students in terms of microstructure and processing.

Part Two: Polymers

The following are abbreviated handouts used for the polymer experiment.

Time Dependent Properties of Polymers

Task: To qualitatively examine the effect of strain rate on the mechanical properties of several thermoplastic polymers and relate those properties to microstructural behavior.

Materials: Three polymer samples.

- Silly Putty
- Six Pack Rings
- Nylon cable tie
- Splat Ball

Group member roles: For this work identify a recorder and a runner for your team. Their responsibilities are:
Recorder - Record all group observations on the supplied sheet.
Runner - Act when needed according to the following instructions.

Instructions:

1. Have runner acquire one of each type of specimen for the group.
2. Silly Putty
    - Each person should determine the effect of strain rate on the mechanical behavior of silly putty by pulling slowly, pulling quickly, and throwing down on the floor.
    - As a group describe (and record) the behavior in engineering terms.
3. Six Pack Rings
    - Each person should pull one loop of the rings very slowly and pull one loop of the rings very rapidly to failure.
    - As a group, describe (and record) the mechanical behavior using engineering terms.
4. Nylon cable ties.
    - Each person should pull one cable tie loop very slowly and pull one cable tie loop very rapidly to failure.
    - As a group, describe (and record) mechanical behavior using engineering terms.
5. Splat balls.
    - Each person should throw the Splat ball on the table top.
    - As a group, describe (and record) mechanical behavior using engineering terms.
5. As a group, Deduce and **Justify**:
    - What happens in the microstructure to produce the behavior in each case. (Are the materials thermoplastics or thermosets?, What are the molecules doing?)
    - What type of polymer would be a good choice for six pack rings. (What are the important criteria and how does your choice meet those criteria?)

Recorder Sheet

1. Description of the mechanical properties (qualitative) of the specimens with emphasis on the effect of strain rate
    - Six Pack Rings
    - Silly Putty
    - Nylon Cable Ties
    - Splat Ball
2. Deductions regarding microstructure

Specimen	Microstructure	Justification
Six Pack Ring		
Silly Putty		
Cable Ties		
Splat Ball		

3. Material selection for six pack rings (and why?).

Instructor Notes:
The students really enjoy throwing the Splat balls and silly putty. Consequently, the learning process can quickly degenerate into pure fun without some gentle external pressure and a time deadline.

At the time of this experiment, the students have had lectures on the difference between thermoplastic and thermosetting polymers. As part of this they have heard about the microstructure, deformation mechanisms and the consequent time- and temperature-dependent behavior (viscoelasticity). This experiment allows the students to experience extremes of polymer response with parts that they recognize and can manipulate.

Silly Putty is a time-honored demonstrator of the coupling of large scale viscous response (creep) under slow strain rates and highly elastic response (and "brittle" fracture) under faster rates. The student's task is to recognize that the elastic response results from the "entropy spring" of chain coiling and uncoiling with relatively little internal friction, while the viscous response results from relative chain sliding.[3]

The polyethylene **six pack rings** are also show the effect of strain rate clearly with significant drawing behavior at slow strain rates and little deformation at high strain rates.[3] Note that many of the newer six pack rings are designed with notches to reduce animal strangulation, and will not draw as expected. The increased opacity of the stretched portion of the rings allows the instructor to discuss crystallinity. The use of a heat gun to reverse the process can reinforce the concept of the aligned, crystallized neck reverting to a less aligned and more amorphous original state.

The **nylon cable ties** reinforce the notion that viscoelasticity of the stronger engineering alloys operating below their glass transition temperature may present more subtly and the effect may be missed in casual examination.[4] If the cable ties have about the same cross-sectional area as the six pack rings, the difference in tensile strength between a low density polyethylene and nylon is also apparent.

Splat toys flatten significantly when thrown against a surface and then slowly recover to original shape. They are included because of their ability to fully recover after large scale deformation. These are available at K-Mart as Splat Bunnies and Splat Trolls. American Science and Surplus (3605 Howard Street, Skokie, IL 60076, 708-982-0870) sells them as Sticky Balls for less than $2 apiece. Again we have an example of a time dependent entropy spring, but this time in a lightly cross-linked polymer that allows the shape recovery. The students generally do not make the connection between the large scale recovery of rubber bands and the recovery of the Splat balls.

In-class discussion following the polymers experiment centered on the design challenges and the engineering applications of viscoelastic materials. Special emphasis was placed on design for creep and applications of energy damping.

Conclusions:

Spending class time in this way was valuable for several reasons. Student evaluations indicated that the majority of the students found the in-class, hands-on work very helpful and recommended that use of such labs be increased in the future. Some students still resisted working in teams, but many also found that aspect helpful. In particular, by observing the groups at work, I was able to see more clearly holes in the student's knowledge, and the problems students were having integrating that knowledge. Finally, the equipment used is relatively inexpensive and easily available.

References:

1. Johnson, D.W., Johnson, R.T., Smith, K, A., *Active Learning: Cooperation in the Classroom*, Interaction Book Company, Edina, MN, 612-831-9500.
2. Newey, C. and Weaver, G., eds.: *Materials Principles and Practice*, Butterworths, London, 1990, pp. 310-311.
3. Ibid, pp. 314-316.
4. Weidman, G., Lewis, P., and Reid, N., eds.: *Structural Materials*, Butterworths, London, 1990, pp. 220-224..

DIELECTRIC ANALYSIS FOR POLYMER PROCESSING

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DIELECTRIC ANALYSIS FOR POLYMER PROCESSING

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ABSTRACT

The cure cycle for an epoxy resin is investigated using a system for monitoring frequency- and temperature-dependent dielectric properties. The dielectric analysis system is computer controlled and features a comb capacitor small enough to be imbedded in a sample of material. The system allows for monitoring of dielectric storage and loss constants for frequencies spanning 5 orders of magnitude, with the temperature of the sample controlled independently. In addition, the dielectric analysis system may be used to study cure mechanisms and glass transition temperatures.

Key Words: cure cycle, dielectric properties, epoxy resins, polymeric materials processing.

Prerequisite Knowledge: General information about polymer structure and synthesis, basic AC circuit theory, definitions of various dielectric properties.

Experimental Objective: To evaluate the cure state of an epoxy resin for various cure profiles using dielectric analysis.

Learning Objectives: After completing this experiment, the student should be able to

1. describe the dielectric response curve as a function of cure time for an epoxy resin.
2. explain the dielectric response curve very generally in terms of the molecular changes during the cure cycle.

INTRODUCTION

The epoxy resins form an important class of thermosetting polymers with high adhesive strength, low shrinkage, good chemical resistance, and excellent mechanical and electrical properties. While many commonly used epoxy resins rapidly crosslink at room temperature when mixed with a curing agent, others require elevated temperatures for cure and may be shipped from the manufacturer with the curing agent premixed.

Epoxy resin manufacturers provide data sheets with recommended cure cycles, typically obtained by evaluating the state of cure by using thermal techniques such as differential scanning calorimetry (DSC) with small specimens. However, the optimum cure cycle may vary depending upon the desired degree of cure for a particular manufacturing application, and may be difficult to determine via thermal techniques due to the size of the product. While an appropriate cure cycle for the product may be identified by thermally cycling the epoxy resin and then conducting post-cure mechanical and electrical tests, this approach may require many attempts before the optimum cure cycle is determined. Therefore, to optimize the curing process efficiently, it is desirable to have an in-situ analysis technique capable of identifying the onset and completion of the crosslinking reactions regardless of specimen size.

Electrical measurements have long been used as an alternative to thermal techniques to estimate the extent of crosslinking [1] in epoxy resins, and the dielectric properties have been found to be a useful indicator of properties such as viscosity, density, and index of refraction for materials during chemical reactions [2]. In particular, measurement of conductivity has been used to monitor the cure processes in thermosetting polymers.

In the classical experimental arrangement shown in Figure 1, the epoxy resin is placed in a capacitor (with known empty capacitance C_0), an alternating voltage V is applied, and the resulting current I is measured. The "resistive" component of the current I_R (that part which is in phase with the voltage and can therefore cause energy dissipation)

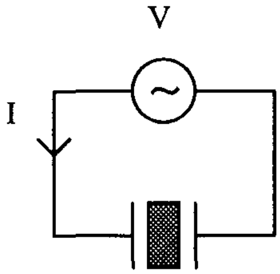

Figure 1. Simplified Circuit for Dielectric Analysis

is given by

$$I_R = \omega C_o \varepsilon'' V \tag{1}$$

where ω is the frequency of the applied alternating voltage and ε'' is the dielectric loss factor [3]. This may be rewritten as:

$$\varepsilon''\omega = I_R / C_o V. \tag{2}$$

The measured current in a polymer has both dipolar and ionic contributions. Dipolar current is due to the motion of segments of the polymer chain with dipolar charge distribution when an electric field is applied. The ionic contribution, on the other hand, results from the migration of charged molecules through the material. The dipolar and ionic currents vary significantly as a function of temperature and the degree of crosslinking. However, if isothermal measurements are made the current (and therefore $\varepsilon''\omega$) will stabilize after an appropriate curing time. The current will, however, vary somewhat at different frequencies due to differing ability of the chain segments and ions to respond. Thus the apparent cure time will vary with frequency, and measurements must be made at a range of frequencies to determine the optimum cure time.

One challenge for this type of analysis has been the ability to perform in situ measurements during fabrication of components with designs that do not readily accept a dielectric sensor. This has resulted in the development of the compact, comb capacitor sensor used in this experiment.

INSTRUMENTATION

A diagram of the experimental apparatus is shown in Figure 2. Each component will be discussed below.

Sensor: The sensor used to monitor the dielectric response is a comb capacitor with a tooth size of 0.02 mm and a tooth spacing of 0.03 mm. The electrode is deposited on a glass or plastic substrate. The sensor dimensions are 1 in. by 0.5 in. The small size of the sensor allows it to be imbedded in a small quantity of the material being studied or in a small section of a large component.

Impedance/Gain-Phase Analyzer: The analyzer applies a voltage to the capacitor and measures the resulting current and phase information. In particular, the voltage difference across the sensor and the current through the sensor are measured. The phase difference between the applied voltage and the current is also determined by the analyzer. The analyzer repeats these measurements for each frequency specified by the user, and transmits the results of each measurement to the computer.

Thermocouples: A Scanner and DMM/Scanner combination is used to monitor up to 8 thermocouples. Typically one thermocouple is placed on or near the sample to measure its temperature. Additional thermocouples may be used as desired by the operator.

Computer: All instruments listed above are connected to a computer which is used for controlling the instruments and data collection. The software for this system is a basic-language program written by Dekdyne, Inc. The software features instrumentation control subroutines which allow for user selection of data acquisition interval, total run time, and AC frequencies for analysis. The frequency range of the analyzer used by the authors in from 5 Hz to 1 MHz. The system may scan up to 11 frequencies during data acquisition.

Temperature Controller: A programmable temperature controller, independent of the computer, is used to control oven temperature. This system features a separate thermocouple for sample temperature measurement.

PROCEDURE

Preparation for measuring dielectric properties requires selection of a cure cycle, initialization of the dielectric system, and application of the polymer resin onto the sensor.

Cure Cycle: Cure cycles for polymer resins are provided by the manufacturer, although the specific application may require modification of the manufacturer's recommendations. Arrangements for controlling the temperature of the oven must be made. The authors use a programmable temperature controller which may be programmed prior to the start of the cure cycle and controls the sample temperature for the duration of the cycle. An example of a cure cycle used by the authors to cure Fiberite 977-2 resin is shown in Table 1. This cure cycle is based upon a cure cycle reported by Fiberite Composite Materials [4], which included a 2 °C/min temperature increase followed by a hold of approximately 38 min at 180 °C.

Table 1. Cure Cycle Selected for Fiberite 977-2 Based Upon Reference 4.

Step	Beginning Temperature	Ending Temperature	Duration
1	30 °C	180 °C	71 min
2	180 °C	180 °C	38 min
3	180 °C	30 °C	30 min

Initialization: Initialization of the dielectric instrumentation involves entering into the computer parameters for the cure cycle, such as total time of operation, interval between data collection, frequencies for analysis, and number of thermocouples. The computer program then enters a calibration routine, which involves measuring the signal from the sensor under conditions of having the circuit shorted, having the circuit open, and finally with the sensor in the circuit but open to air. The program then pauses until the operator is ready for data acquisition to begin.

Sample Preparation: For sample preparation, the sensor must be brought into contact with the material to be cured. For liquid epoxy studies, the authors place the sensor at the bottom of a small reservoir. The uncured epoxy is then prepared for curing as directed by the manufacturer and poured onto the sensor. As epoxy resins may be quite viscous, good contact is often not achieved at this point. However, as the sample is heated, it will become less viscous and will spread over the sensor.

Data acquisition: Once initialization is complete, the temperature controller is started and the data acquisition routine of the program is started. For the duration of the cure cycle, the system is automated, and no action by the operator is needed. For analysis, the system sends an AC signal of known current, voltage, and frequency to the sensor, and measures the voltage across the sensor, resulting current, and phase difference on the opposite terminal. From these data, the capacitance and impedance of the capacitor may be calculated, and, in turn, the dielectric storage and loss constants may be found. These data are recorded at intervals (typically 60 sec) throughout the cure cycle, and may be displayed graphically during acquisition or stored and plotted later.

RESULTS AND ANALYSIS

An example of the results is shown in Figure 3, where $\varepsilon''\omega$, (the dielectric loss constant multiplied by the frequency) of Fiberite 977-2 epoxy resin is plotted as a function of time during the cure cycle. As the temperature of the resin is raised, $\varepsilon''\omega$ increases, indicating an increase in the in-phase current (see eq. 2). During this stage, the current in the uncured epoxy is largely ionic, and the increasing $\varepsilon''\omega$ is an indication that ions are migrating through the material more readily. Furthermore, as the temperature increases, polymerization reactions are producing additional ions.

At the onset of cure indicated in Figure 3, the crosslinking reactions consume ions causing the ionic contribution to the current to begin to decrease. These reactions cause the formation of a molecular network, segments of which may undergo small scale motion as a result of their dipolar charge distribution. Thus, the dipolar contribution to the conductivity increases to partially offset the ionic decline as the cure state advances.

Eventually, dipolar contributions become the dominate conduction mechanism. Presumably, the ionic current contribution will become negligible as the network becomes more complete. Then a value of $\varepsilon''\omega$ is reached that is due solely to dipolar motion. Therefore, the flatness of the $\varepsilon''\omega$-time curve after a dwell time at the cure temperature should indicate the completeness of cure. Dipolar contributions are dominant as the temperature is lowered at the end of the cure cycle.

EQUIPMENT LIST

The dielectric apparatus as used by the authors and available from Dekdyne has the following components:

Dielectric Sensors

Schlumberger 1260 Impedance Gain/Phase Analyzer

Keithley 199 System DMM/Scanner

Keithley 705 Scanner

IBM PS/2 Computer

INSTRUCTOR'S NOTES

Equipment: Substitute components may be used. The scanner system used for thermocouple reading was chosen for the purpose of reading multiple thermocouples. Single thermocouple readers that may be interfaced with a computer are available. The major cost of this system is the gain/phase analyzer. Other impedance analyzers are available from Hewlett Packard amongst other vendors.

Other applications: An inflection in the loss constant curve, indicative of the glass transition, is characteristic of dipolar current contributions. The system may therefore be used to measure glass transition temperatures.

REFERENCES

1. Handbook of Epoxy Resins Henry Lee and Kris Neville McGraw-Hill 1967 p.6-13.
2. Epoxy Resins and Composites IV, Advances in Polymer Science 80 K. Dusek, ed. ("Dielectric Analysis of Thermoset Cure" S.D. Senturia and N.F. Sheppard, Jr.) Springer Verlag p.3.
3. Electrical Properties of Polymers A.R. Blythe Cambridge University Press 1979 p. 40.
4. Rheological Behavior of Fiberite 977-2 Resin, Fiberite Composite Materials, Doc # 77-9.

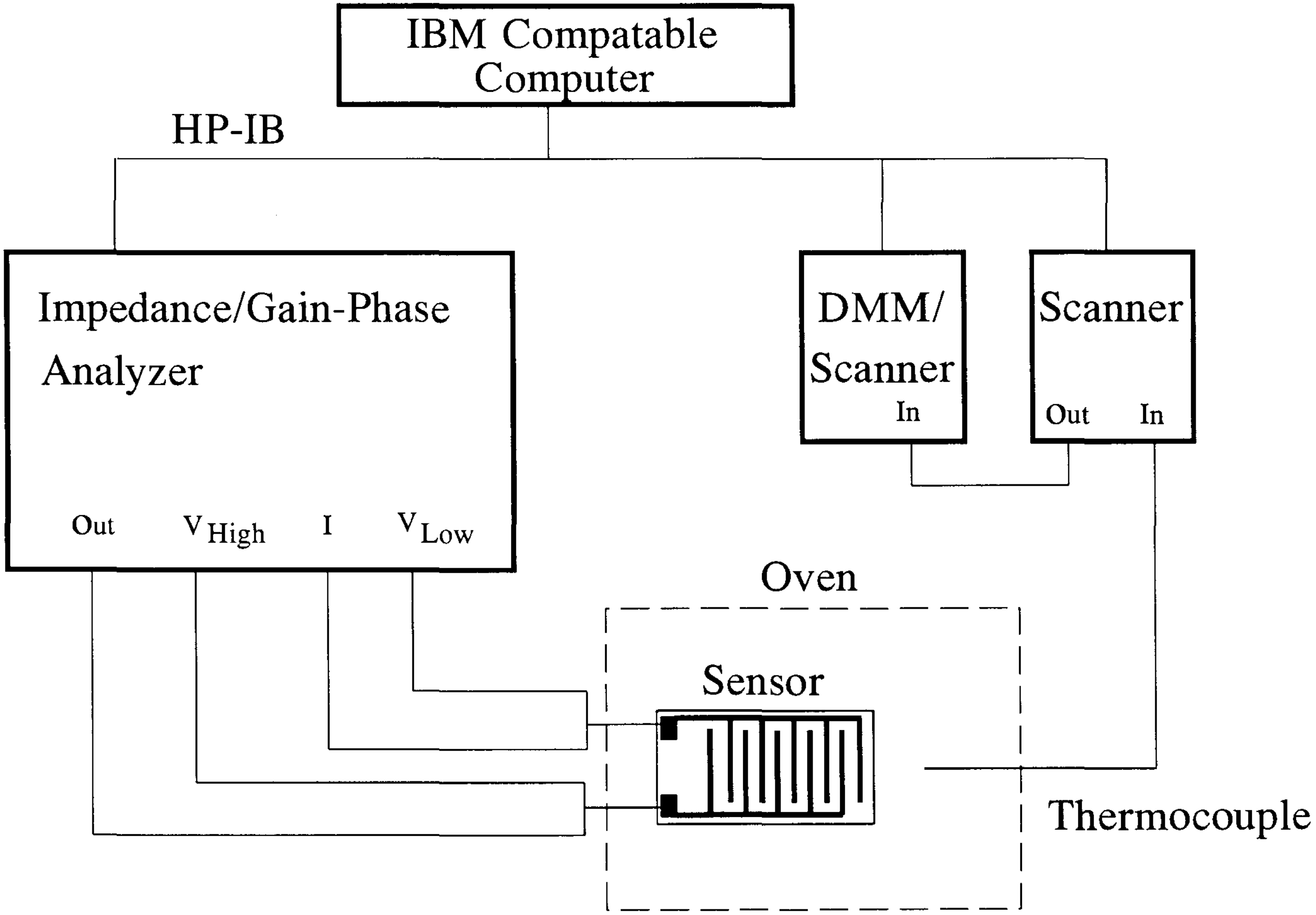

Figure 2. Diagram of AC Dielectric Analysis System.

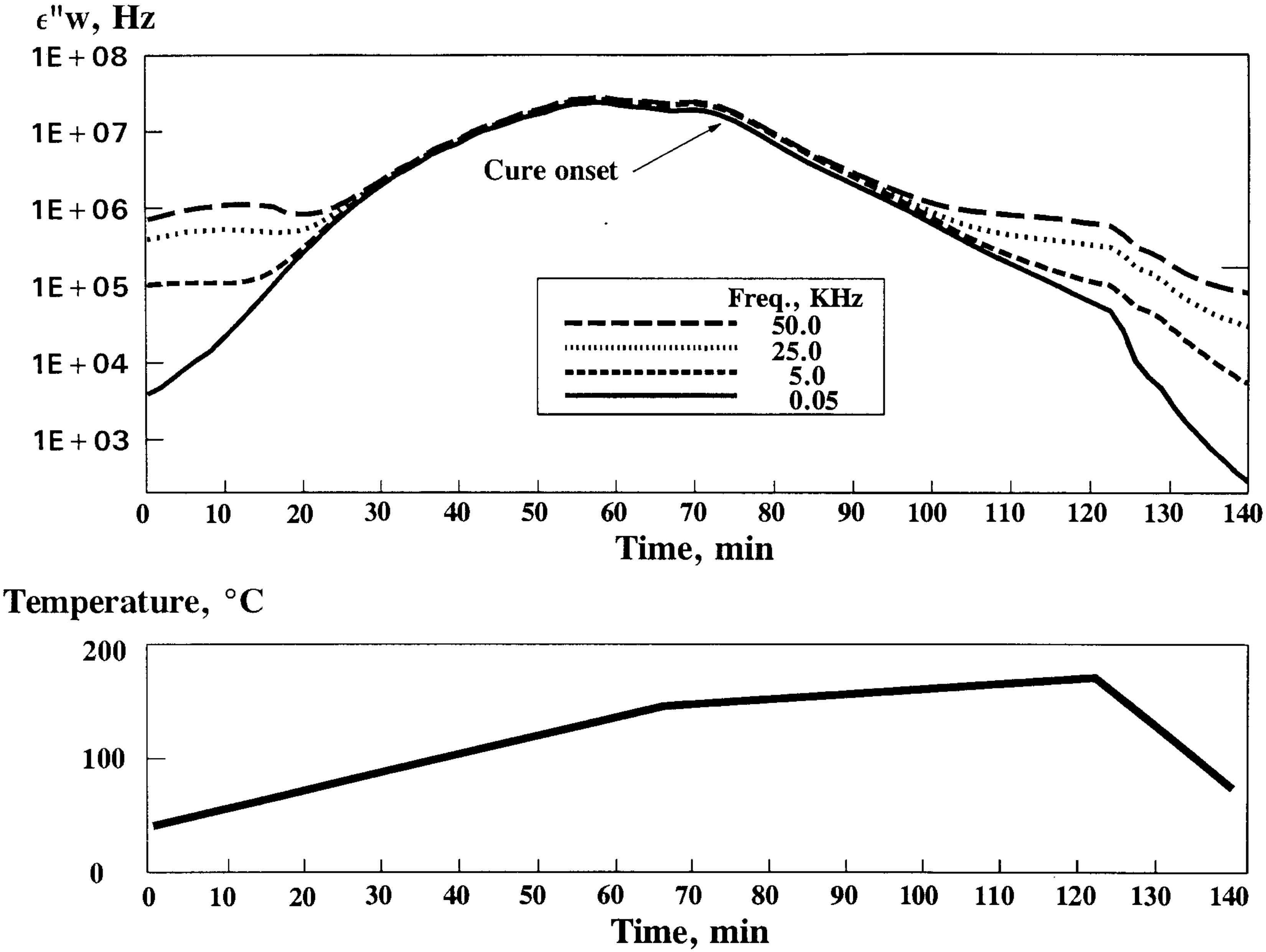

Figure 3. Dielectric Response for Fiberite 977-2 Resin.

CRYSTAL MODELS FOR THE BEGINNING STUDENT: AN EXTENSION TO DIAMOND CUBIC

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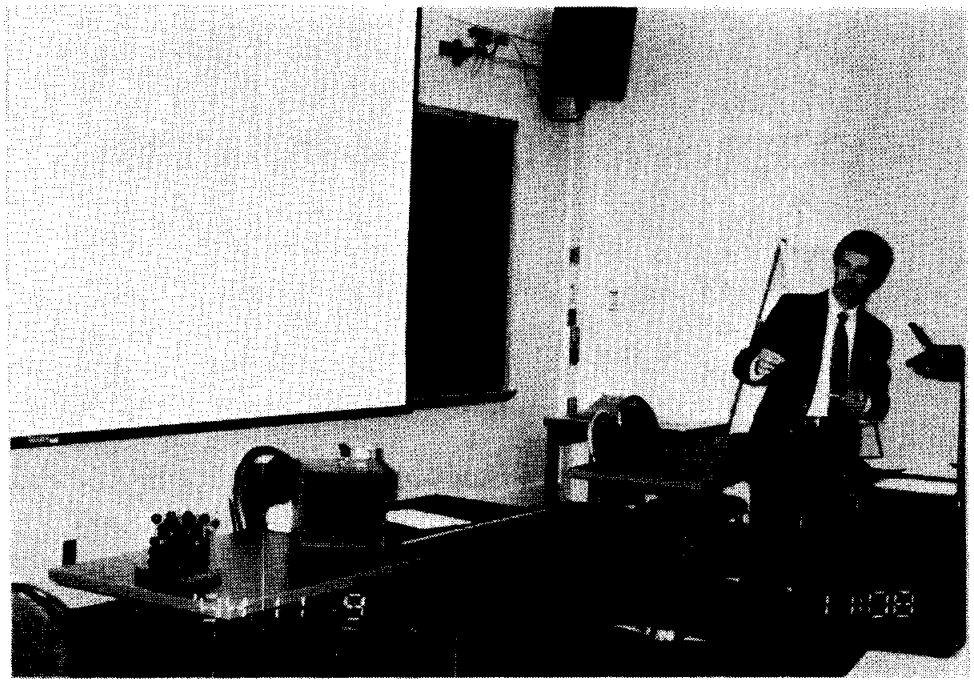

Crystal Models for the Beginning Student: An Extension to Diamond Cubic

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Key Words: Crystal, structure, model, diamond cubic

Prerequisite Knowledge: Knowledge of elementary crystal structures.

Objective: To build a model of the diamond cubic structure on the same template used for construction of the body-centered cubic structure.

Equipment:

1. Base template measuring 6" x 6" x 1" made of wood or Micarta with 3/32" holes drilled 1/2" deep as shown in Figure 1.
2. Stainless steel rods 3/32" in diameter and 6-1/2" in length and inserted into the holes in the base.
3. Wooden spheres (at least 95 to make eight unit cells) nominally 1" in diameter with 1/8" diametral holes. These are available from Klinger Educational Products Corp., College Point, New York (Phone 1-800-522-6252). These spheres actually measure 1.025" in diameter which has been accounted for in the spacing of the rods.

Introducton:

This paper gives an adaptation of the crystal models developed by F. Xavier Spiegel and presented at the National Educators Workshop: Update '90[1]. The models were originally designed to give students a better visualizaton of directions, planes, and three-dimensional relationships in face centered cubic (FCC), body centered cubic (BCC), and hexagonal close packed (HCP) crystal structures. With increased student exposure to the growth and characterization of semiconductive materials, a representation of the diamond cubic structure is also desirable. Simply enough, a [100] orientation of this structure can be built on the BCC template.

Construction:

The diamond cubic structure is built upon the same template as given in Reference 1 for the body centered cubic structure (Fig. 1) and is based upon the face centered cubic structure with half of the tetrahedral interstitial sites filled with host atoms. The diamond cubic structure is not a unique lattice because, when second neighbor relationships are examined, the environments of all atoms are not equal. The strucure is treated as an FCC lattice with a basis of two atoms per lattice site, (0, 0, 0) and (1/4, 1/4, 1/4). The first plane of atoms (z=0 as shown in Fig. 2) is typical of the (100) plane of FCC but note that the unit cell has twice the lattice parameter (a) as the BCC unit cell normally constructed on this template. The second layer (z=a/4 as shown in Fig. 3) has atoms placed in half of the FCC tetrahedral intersitial sites at unit cell locations (1/4, 1/4, 1/4) and (3/4, 3/4, 1/4). Continuing with the third layer (z=a/2 as shown in Fig. 4), typifying the midplane of the FCC structure, the atoms are placed at (1/2, 0, 1/2), (0, 1/2, 1/2), (1/2, 1, 1/2), and (1, 1/2, 1/2). The fourth layer (z=3a/4 as shown in Fig. 5) puts atoms over the unoccupied tetrahedral sites, namely (1/4, 3/4, 3/4) and (3/4, 1/4, 3/4). The fifth and final layer of the unit cell repeats the first layer; however, the corner atoms at (0, 0, 1) and (2, 2, 1) will need support as they will not be in direct contact with any of the previously placed atoms. This dictates the insertion of spacers constructed from straws, coffee stirrers, or tubing, of length equal to (a-2r). Using our template based on spheres of diameter 1.025" with a diamond cubic lattice parameter of 2.368", the spacers should have a length of 1.343" and should be placed over the rods in the locations denoted in Figure 2. Figures 6 and 7 show the completed model from the top and side, respectively.

Notes to the Instructor:

The students can be presented with such questions as:

What is the packing factor for this structure?
The answer is half of that for BCC (0.34) as it has 8 atoms per unit cell (four times as many as BCC) but occupies eight times the volume.

Why does such an open structure result in such a strong, yet brittle, material?
The answer is that this structure is composed of Group IV elements whose electrons prefer strong, highly directional covalent bonding (hence the four-fold coordination) as opposed to metallic bonding where the valence electrons are not closely bound to each host atom. The metallic bond may result in higher coordination, but the atom-atom (ion-ion) bonding is lower and slip can occur before the overall structure fails.

This strucure could be built onto an existing BCC structure to give the students an introduction to the concept of epitaxial growth. In reality, however, BCC carbon does not exist and the substrates which have successfully been used for the growth of diamond (i.e. silicon and molybdenum) primarily develop an intermediate layer of carbide.

Reference:

1. Harris, Jonice S. and Jacobs, James A., eds.: *National Educators Workshop: Update '90,* NIST Special Publication 822, November 1991, pp. 31-44.

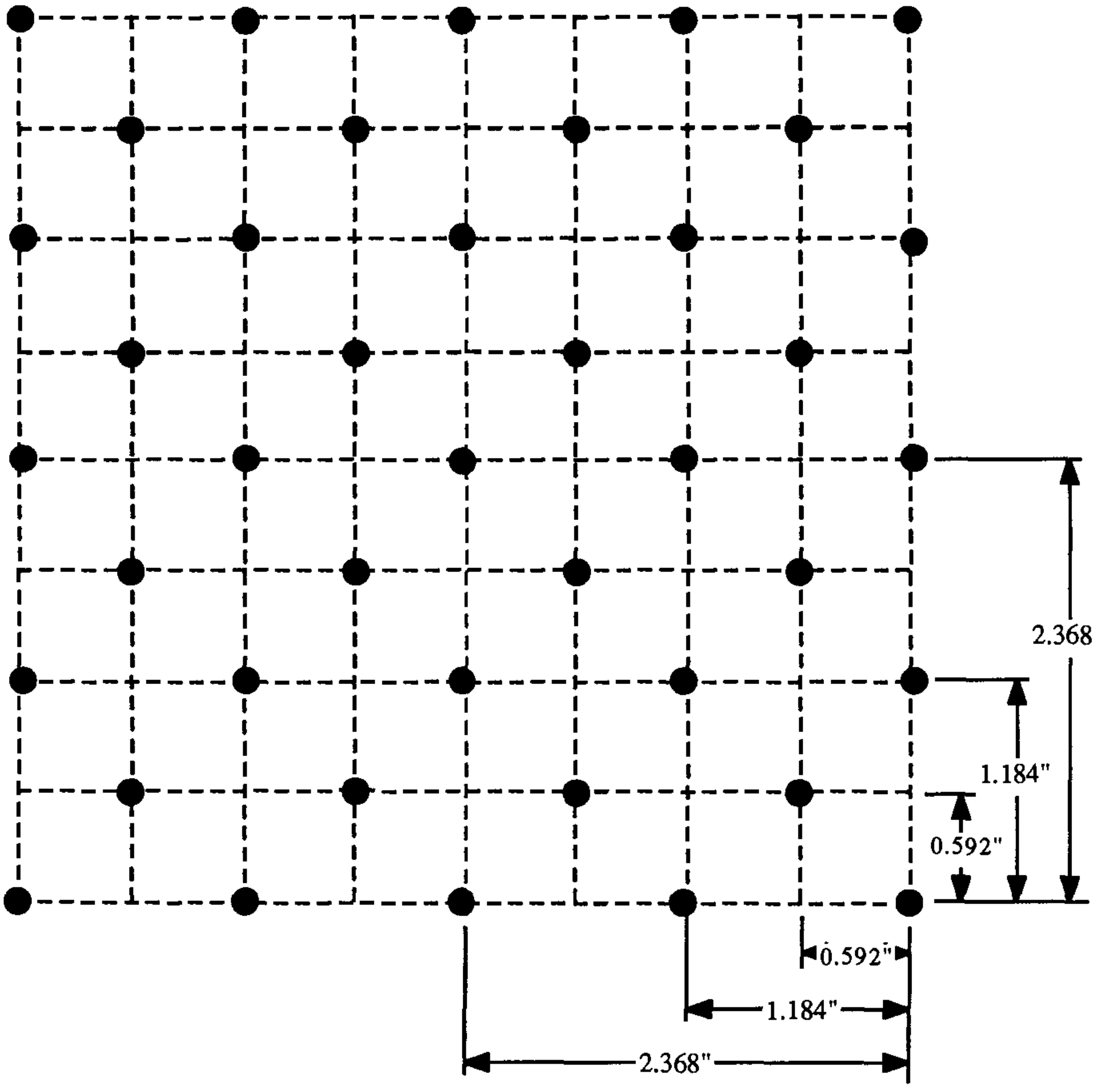

Figure 1. Base layout for the placement of holes for the diamond cubic model

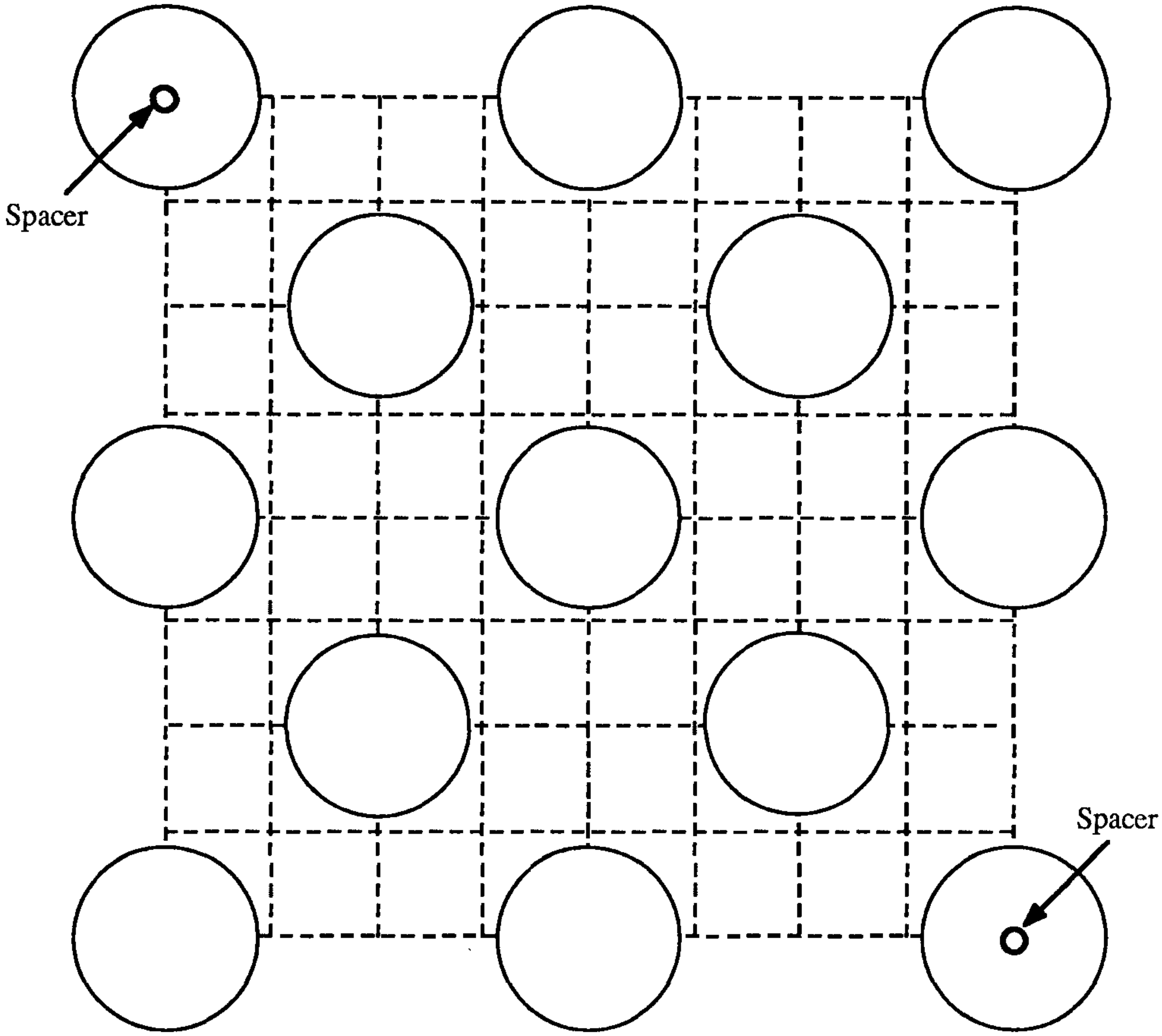

Figure 2. First and fifth layers of atoms (z=0 and z=a). Spacers are used after the first layer.

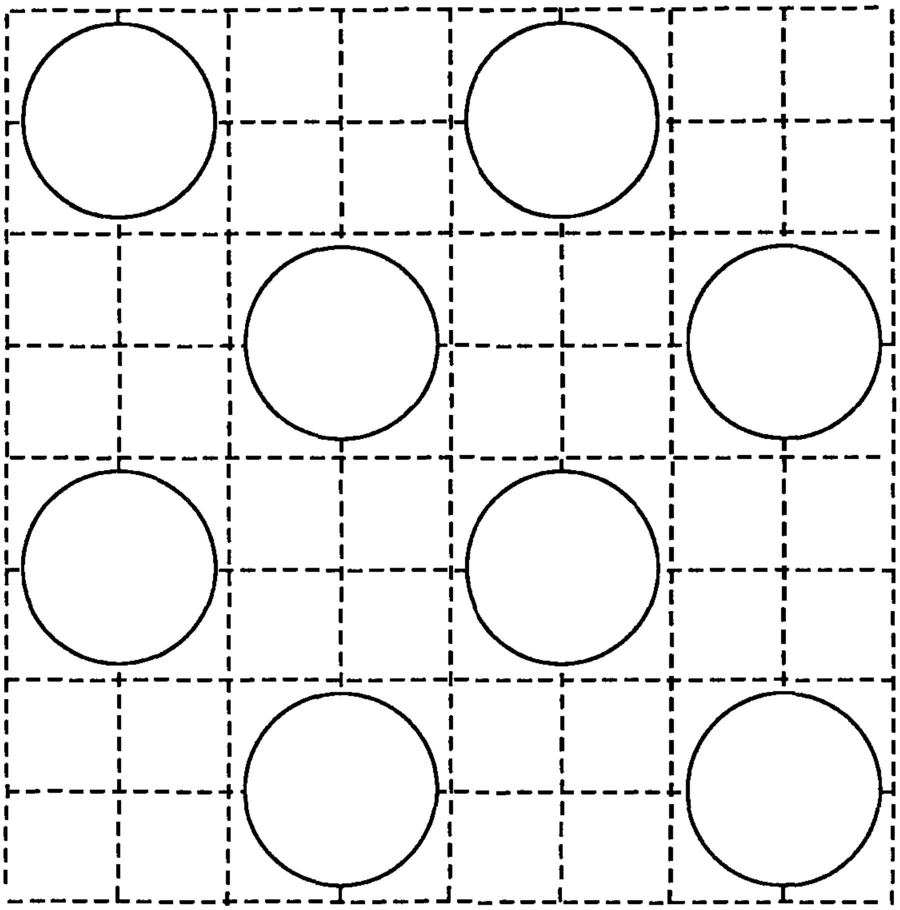

Figure 3. Second layer of atoms (z=a/4)

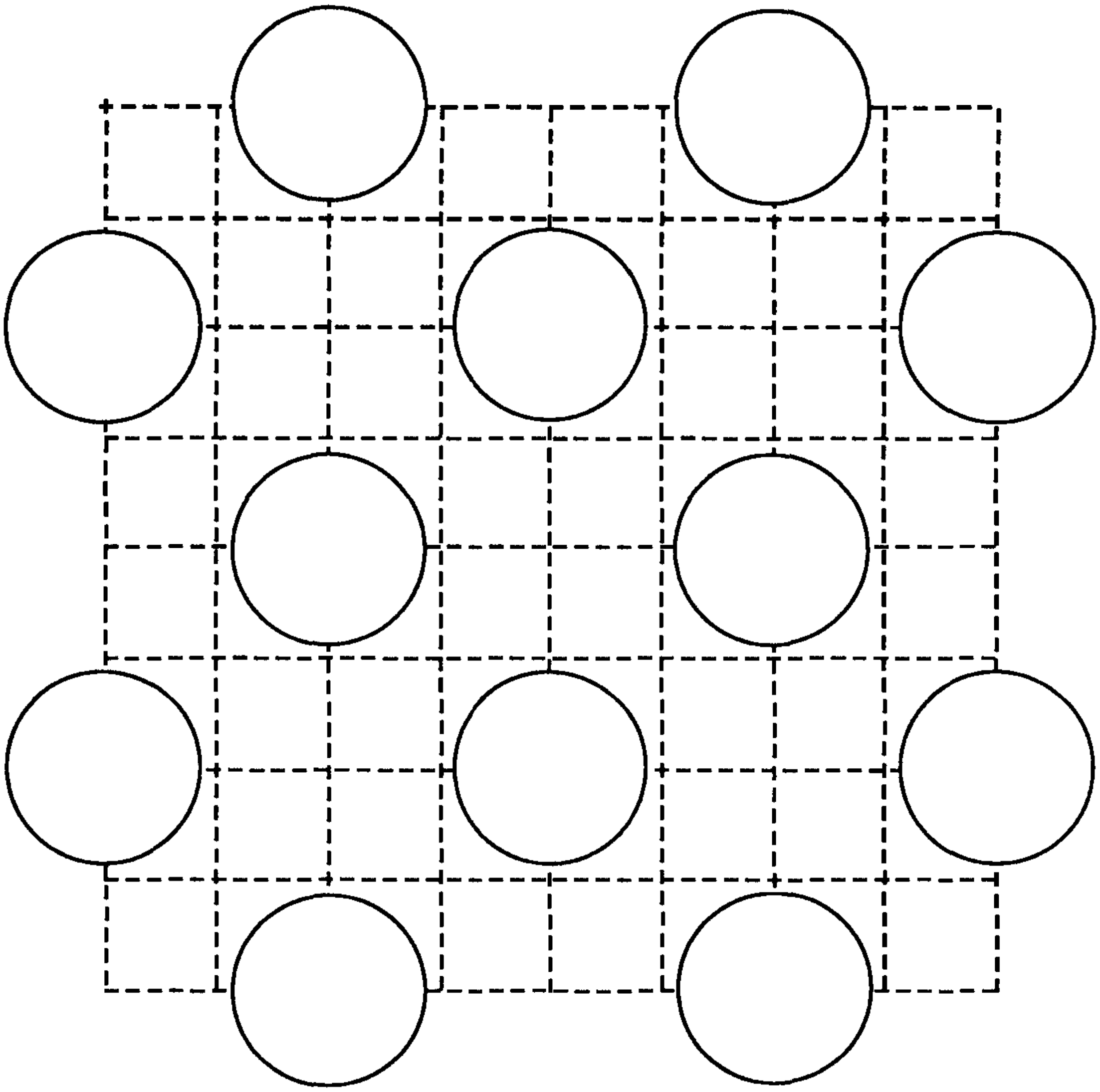

Figure 4. Third layer of atoms (z=a/2)

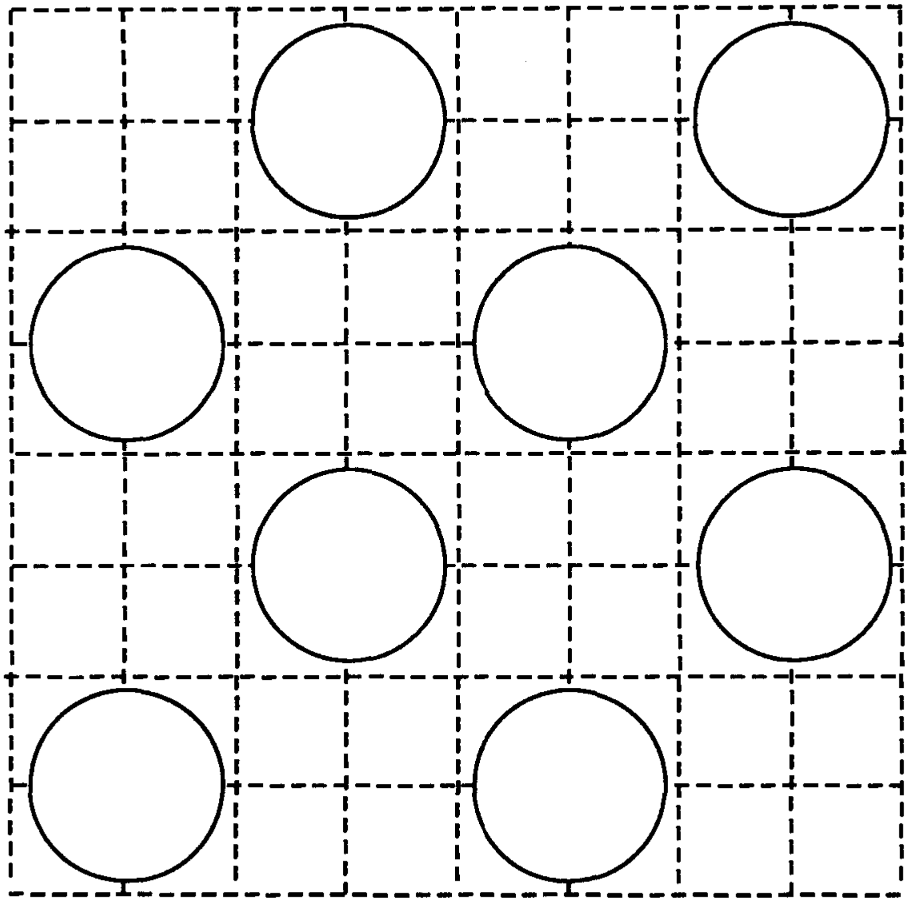

Figure 5. Fourth layer of atoms (z=3a/4)

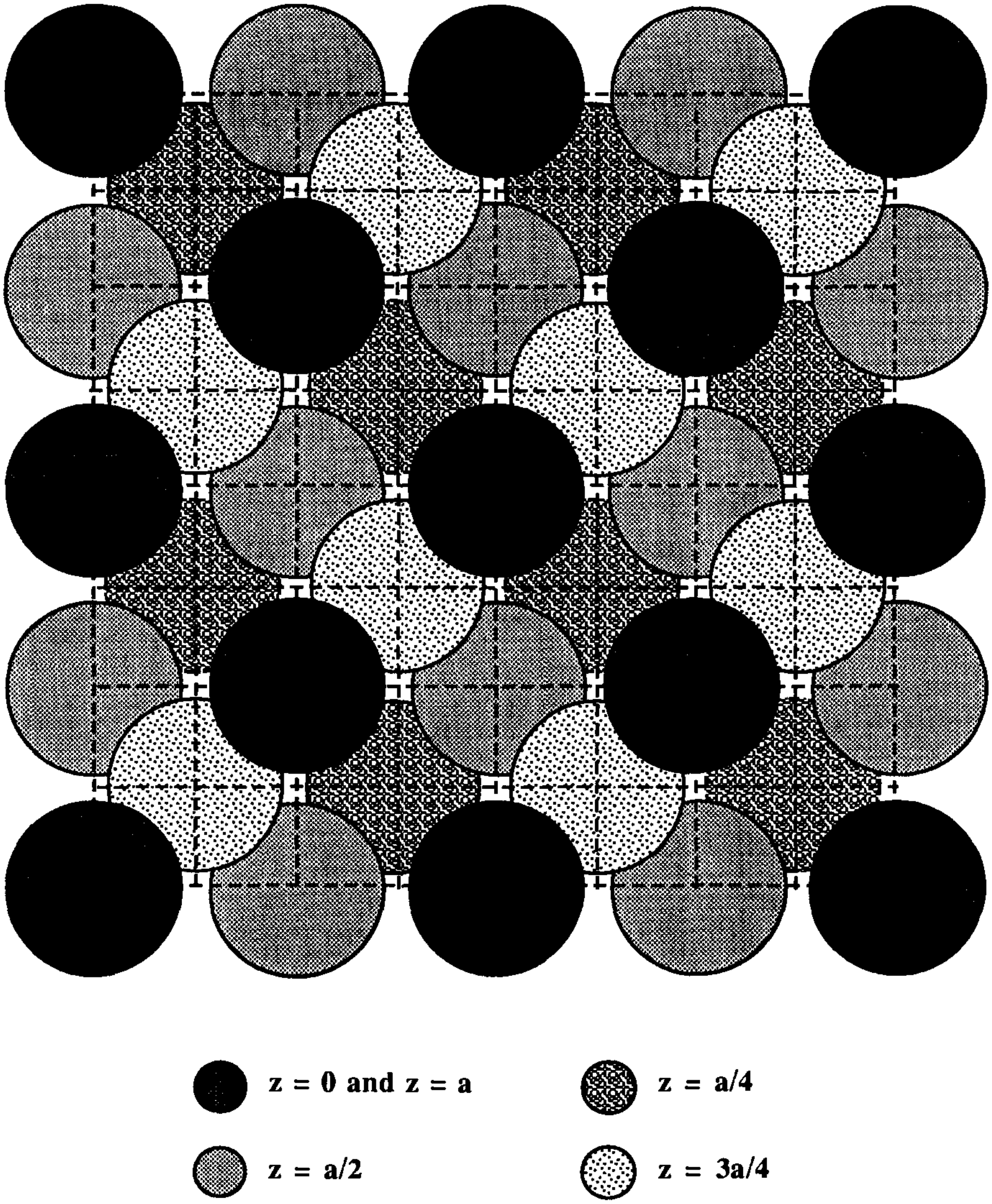

Figure 6. Top view of model

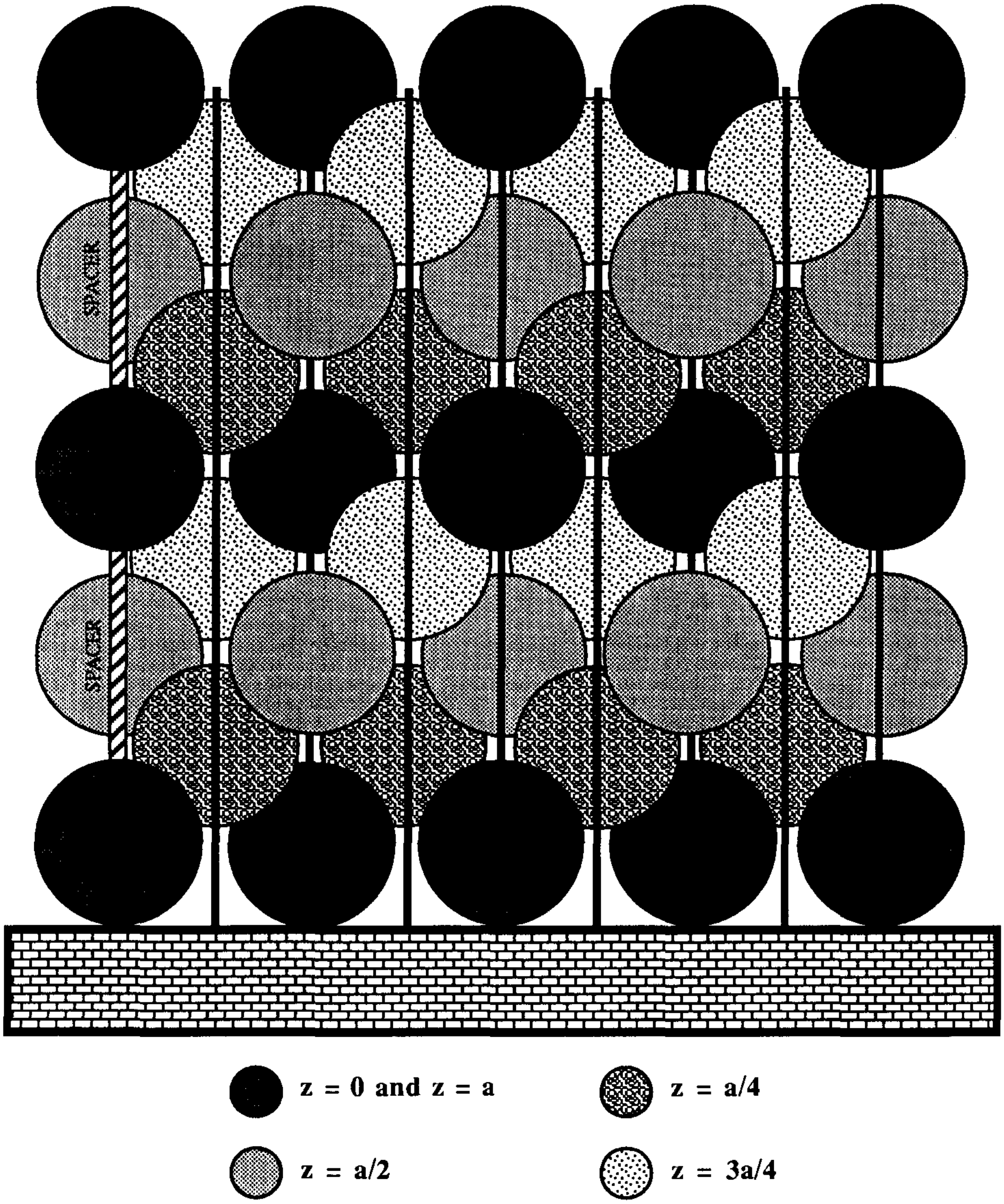

Figure 7. Side view of model with two layers of unit cells

TRANSFERRING KNOWLEDGE TO THE SHOP FLOOR

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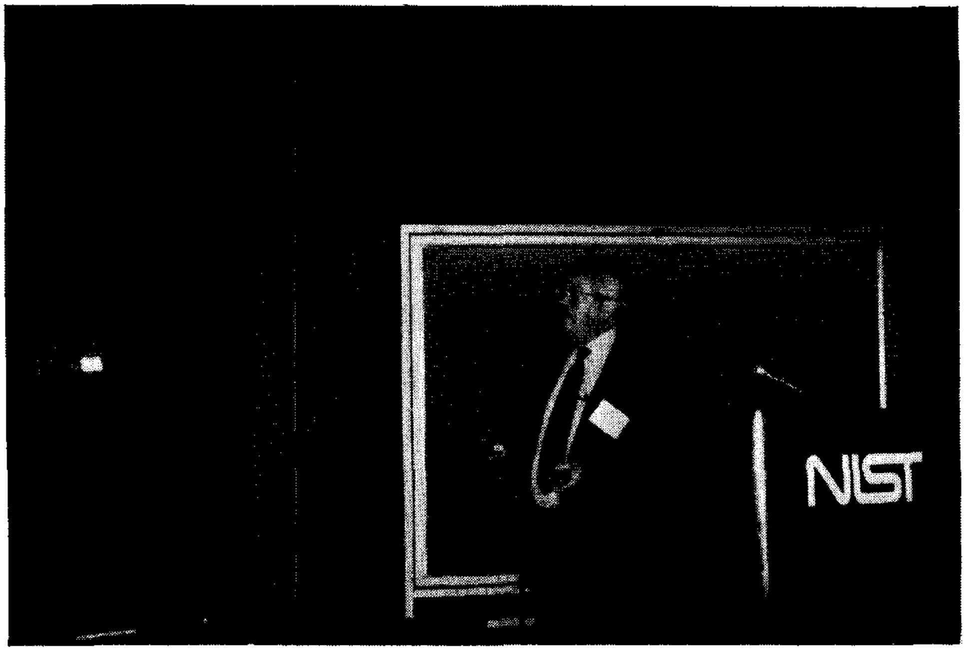
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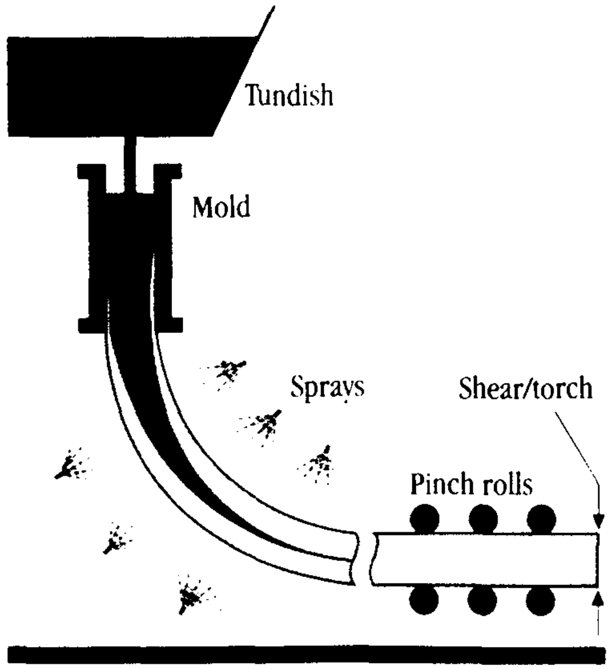

Figure 1 *A schematic of a billet caster.*

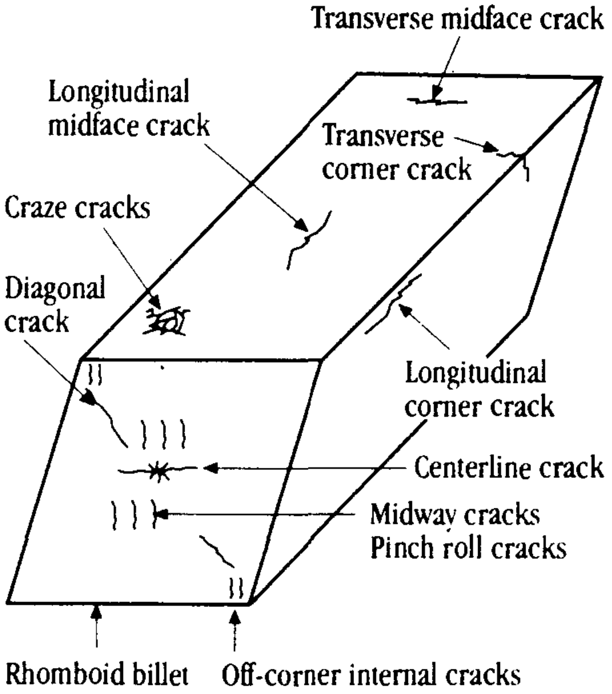

Figure 2 *Schematic of a rhomboid billet showing various types of cracks.*

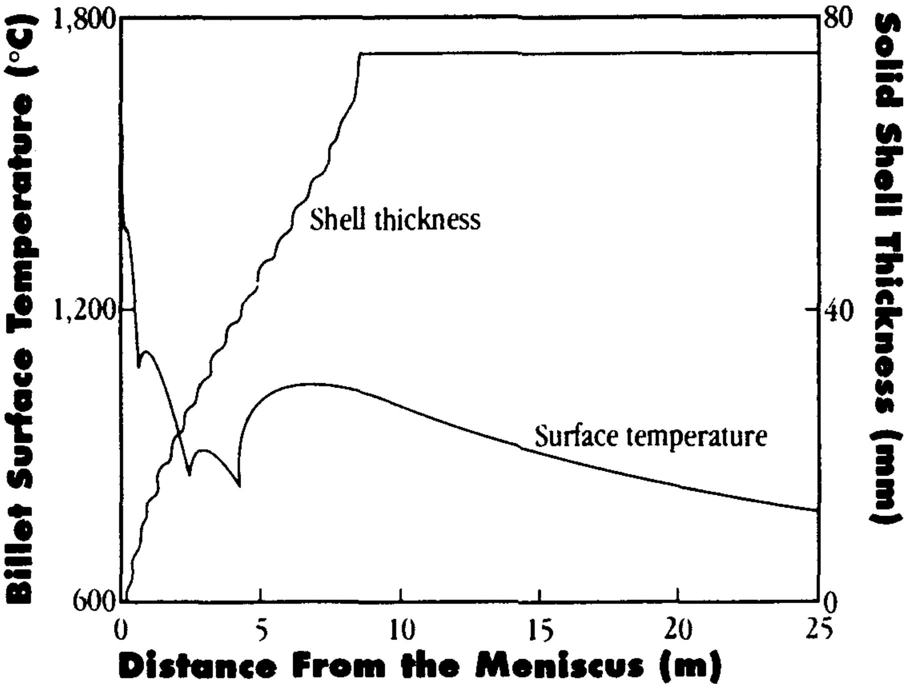

Figure 3 *A typical output of the mathematical model showing predicted solid shell thickness and billet surface temperature profiles (casting speed of 25 mm per second; section size, 150-mm square).*

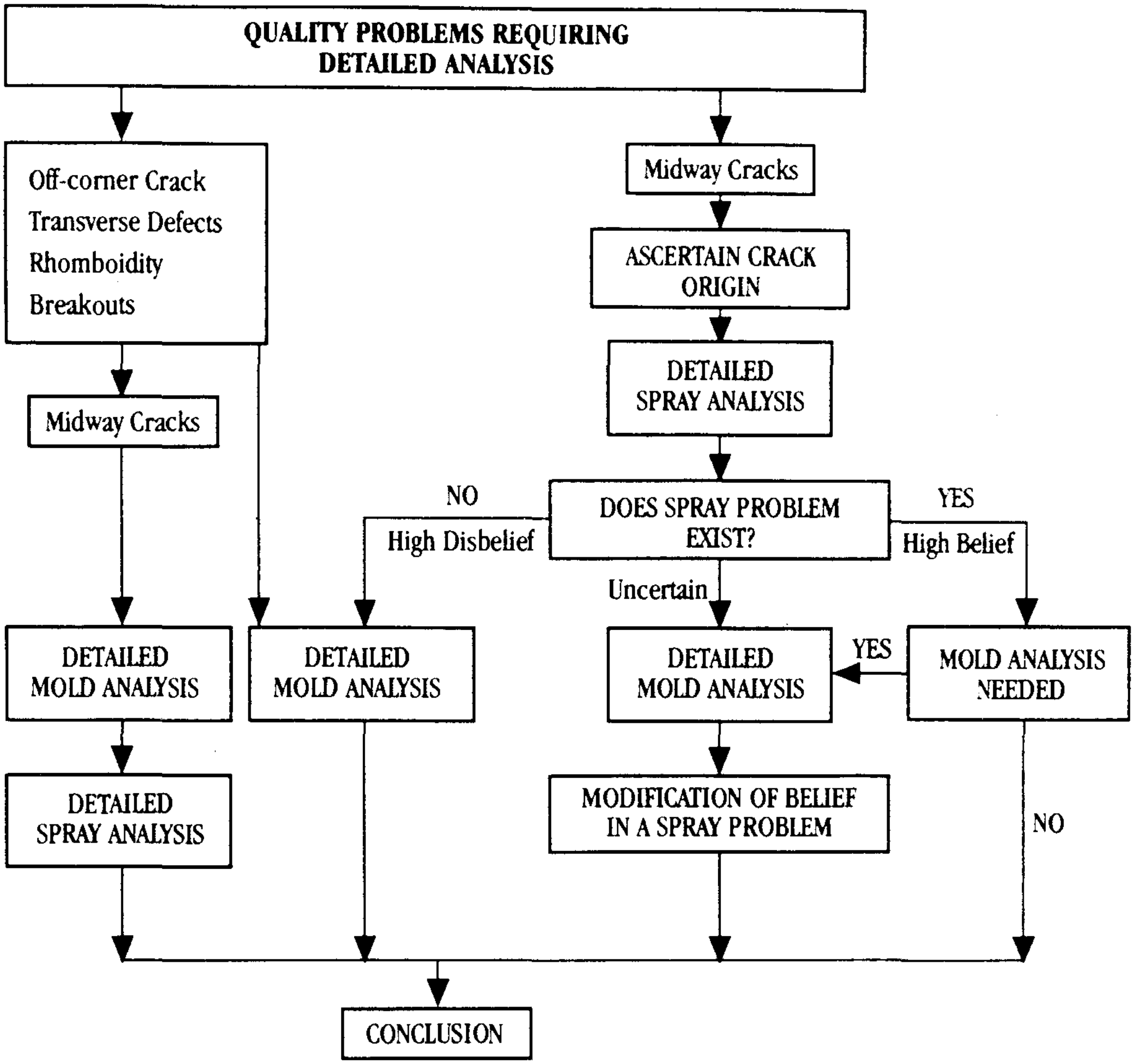

Figure 4 *Scheme for diagnosing quality problems requiring detailed mold and/or spray analysis.*

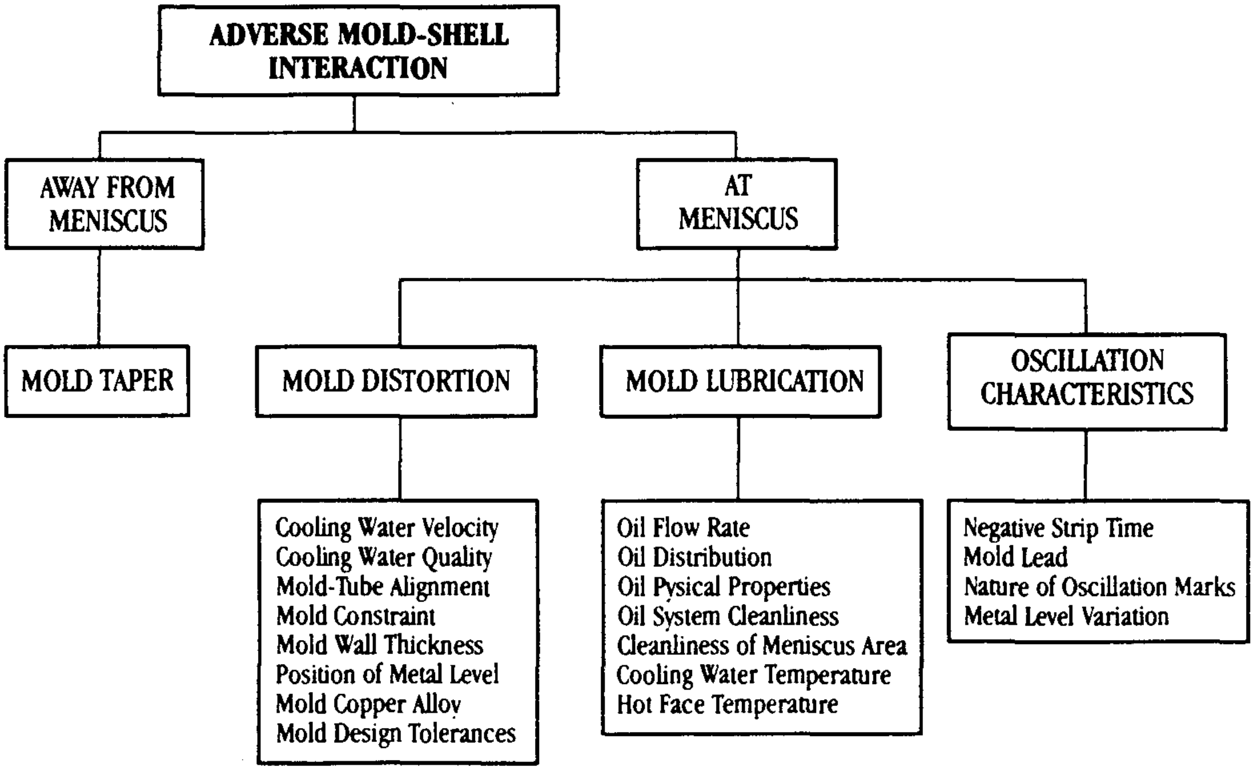

Figure 5 *A general outline of mold-related factors that contribute to quality problems in billet casting.*

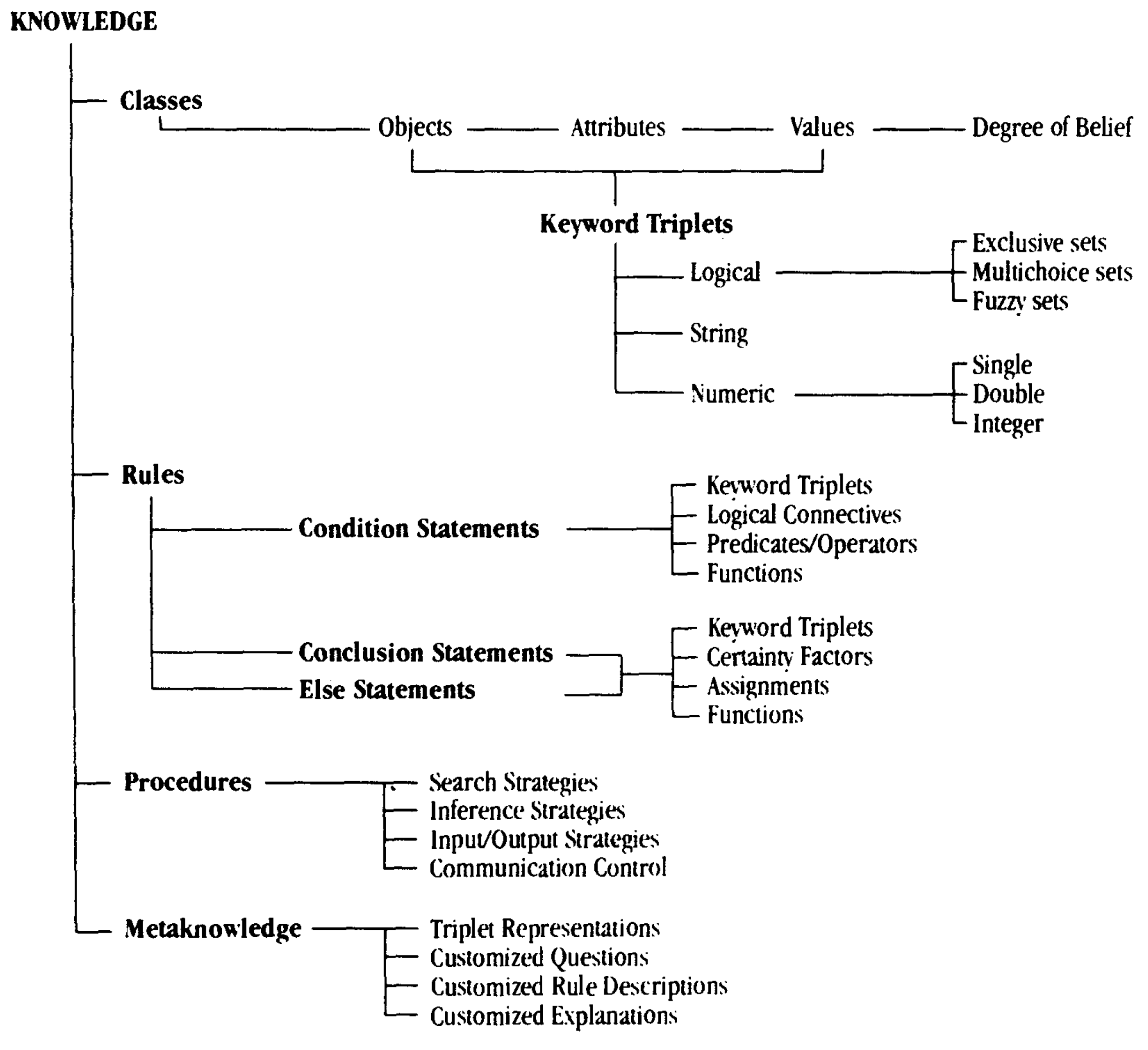

Figure 6 *Knowledge units available in the COMDALE/X development tool.*

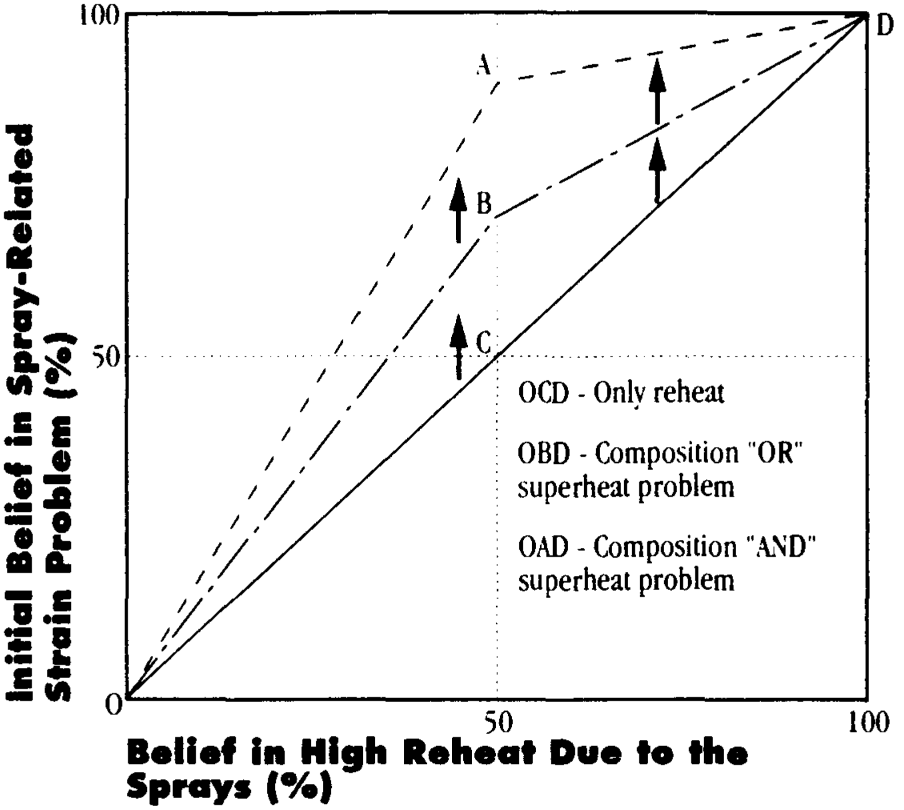

Figure 7 *A schematic illustrating the calculation of degree of belief in a spray-related tensile strain problem from belief in high reheat in the radiation zone, high superheat and composition problems.*

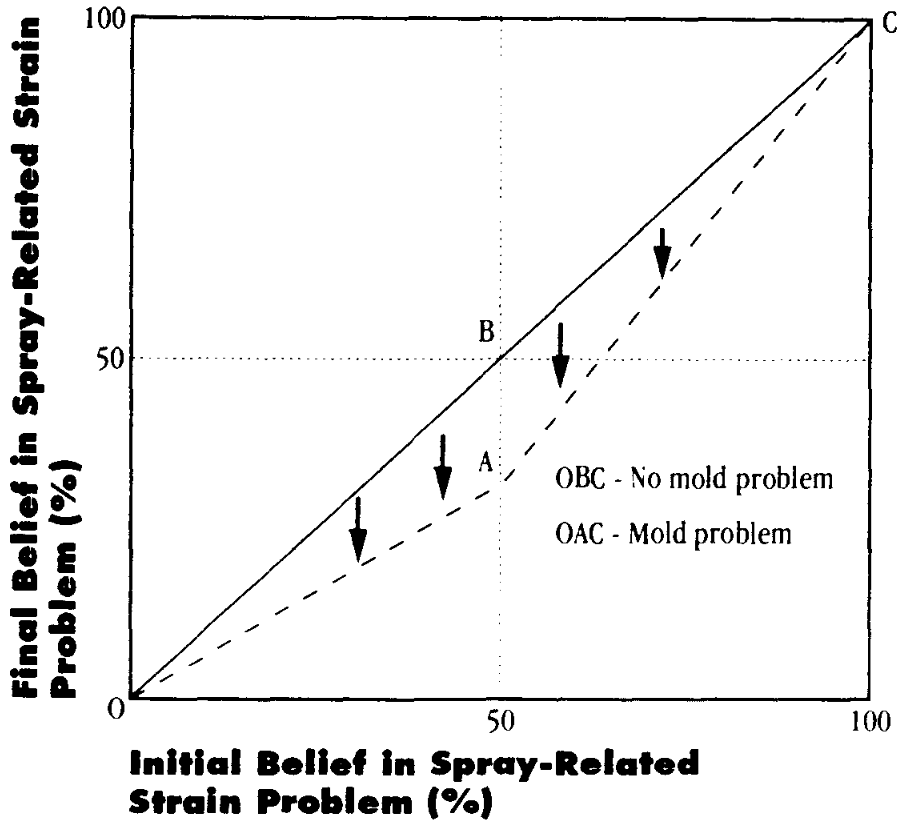

Figure 8 *A schematic illustrating the calculation of final belief in a spray-related tensile strain problem following the detection of a mold disorder.*

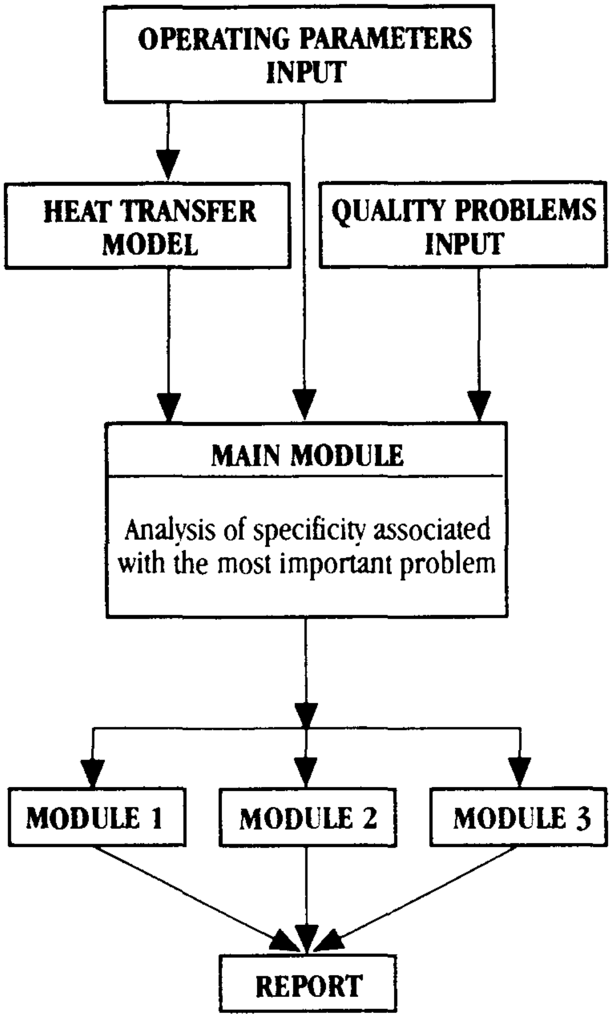

Figure 9 *A general flow sheet representing the structure of the expert system.*

Table I *Internal Cracks in Billet Casting*

Quality Problems	Location	Causes	Influencing Factors
Off-corner crack	Lower part of mold or very close to mold exit	Bulging of the solid shell and hinging at off-corners	Thermomechanical behavior of the mold; adverse mold-shell interaction; deep and nonuniform oscillation marks; steel composition and superheat
Diagonal crack	Spray zone	Nonuniform shell generated by the mold	Thermomechanical behavior of the mold; adverse mold-shell interaction; asynchronous intermittent boiling in the mold; deep and nonuniform oscillation marks; steel composition and superheat
		Asymmetric spray cooling	Poor spray design and maintenance; steel composition and superheat
Centerline crack	Near the point of complete solidification	Sudden decrease in the centerline temperature at the point of complete solidification	Inadequate spray cooling near the point of complete solidification; steel composition and superheat
Pinch roll crack	Close to the pinch rolls	Squeezing on a strand with a liquid core	Excessive pinch roll pressure; steel composition and superheat
Unbending cracks	Close to the point of unbending	Unbending on a strand with a liquid center	Excessive bending strains; steel composition and superheat; high casting speed

Table II *Midway Cracks in Billet Casting*

Locations	Causes	Influencing Factors
Mold exit or in the gap between the mold and the sprays	Reheating of the billet surface	Mismatch between the mold and the sprays due to design or maintenance problem; poor design of cooling jacket near the mold exit; steel composition and superheat
Upper portion of the sprays	Reheating of the billet surface	Poor spray maintenance due to bent or plugged spray nozzles; steel composition and superheat
Lower portion of the sprays or the radiation cooling zone	Reheating of the billet surface due to the sprays	Design and maintenance issues for the sprays; steel composition and superheat
	Reheating of dark overcooled patches generated by the mold	Thermomechanical behavior of the mold; adverse mold-shell interaction; deep and nonuniform oscillation marks; steel composition and superheat

Table III *Surface Cracks in Billet Casting*

Quality Problems	Origins	Causes	Influencing Factors
Transverse crack (and depression)	In the mold	Pulling action on the strand as a result of binding or sticking in the mold	Thermomechanical behavior of the mold; adverse mold-shell interaction; deep and nonuniform oscillation marks; steel composition and superheat
Longitudinal corner crack	In the mold	Reheating of the corner due to a large mold/shell gap and/or Thin and weak shell at the hotter corners	Large corner radius; presence of corner "keyholes," mold-tube alignment; asynchronous intermittent boiling in the mold; deep and nonuniform oscillation marks; thermomechanical behavior of the mold; adverse mold-shell interaction; steel composition
Longitudinal midface crack	In the mold	Excessive reheating of a localized portion of the billet surface; stream impingement on a face	Presence of scratch or gouge marks on the inner surface of the mold wall; misalignment of metal stream
Craze crack	In the sprays	Grain boundary embrittlement due to the presence of Cu, Sn or low melting impurities in steel	High level of Cu, Sn or low melting impurities in steel

Table IV *Summary of Rhomboidity Problem in Billet Casting*

Origins	Causes	Influencing Factors
In the mold and/or the sprays	Nonuniform shell generated in the mold	Thermomechanical behavior of the mold; adverse mold-shell interaction; asynchronous intermittent boiling in the mold; deep and nonuniform oscillation marks; mold-tube alignment; steel superheat
In the sprays	Asymmetric spray cooling	Poor spray design and maintenance; steel superheat

Table V *Summary of Breakout Problems in Billet Casting*

Origins	Causes	Influencing Factors
Close to obtuse-angle corners of the strand	Thin shells generated at the obtuse-angle corners due to asymmetrical cooling in the mold	Thermomechanical behavior of the mold; adverse mold-shell interaction; deep and non-uniform oscillation marks; Mold-tube alignment; steel superheat
Close to transverse depressions or deep oscillation marks on the billet surface	Local reduction in the shell thickness due to the presence of depressions or deep oscillation marks	Adverse mold-shell interaction; deep and non-uniform oscillation marks; thermomechanical behavior of the mold
Close to a weak spot in the shell	Inadequate shell thickness at the mold exit	Insufficient dwell time in the mold; entrapment of slag or scum between mold and billet
Mold overflow	Operator error	Poor metal control

Table VI *Initial Scheme Proposed by the Experts for the Analysis of Midway Cracks Originating in the Radiation Zone*

Stages	Details
1 Input	Distance from the outer tip of the crack to the billet surface
2 Compare	Measured distance with solid shell thickness profile
3 Establish	Location of crack origin in the machine
4 Input	Model predicted billet surface reheat for radiation zone; spray parameters – zone length and water flow rate; high temperature strength of steel – Mn:S ratio; cast structure – superheat level
5 Analyze	Compare input parameters with values specified by the experts
6 Correlate	Generation of midway crack with the operating parameters
7 Conclude	Conclusions and justification

Table VII *Definition of a Fuzzy Set for the Adjective "High"*

Radiation zone reheat temperature (°C)	0	50	75	100	150
Degree of belief in a high reheat (%)	0	25	50	75	100

Table VIII *Examples of the Successful Diagnosis of Quality Problems*

Company	Comments on the Analysis
A	The system related a problem with rhomboidity to plugged spray nozzles. The operators solved this problem by improving the spray cooling water quality.
B	Off-corner cracks were found to originate near the mold exit. The system suggested a mismatch between spray and mold heat extraction rates due to a large gap between the mold exit and the first spray nozzle.
C	The heat transfer model calculated a high reheat in the radiation zone. Midway cracks were seen in the radiation zone. The cause of this high reheat was a short spray zone.

UNDERSTANDING PHASE DIAGRAMS

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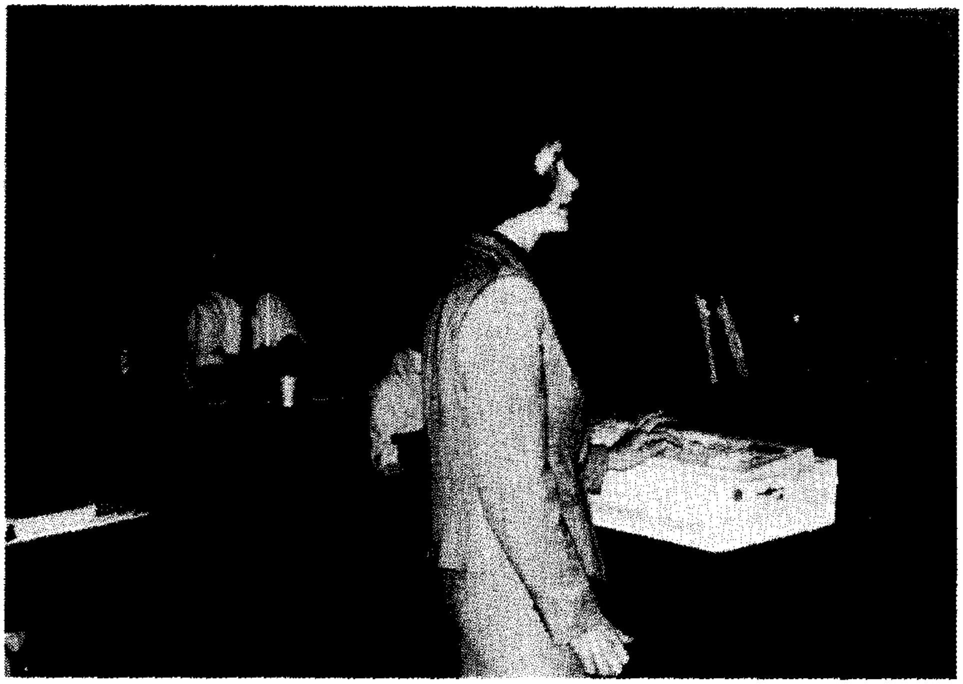

Understanding Phase Diagrams

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Key Words: phases, liquidus, solidus, solvus, eutectic reaction

Prerequisite Knowledge: General knowledge of phase transformations as affected by temperature and composition.

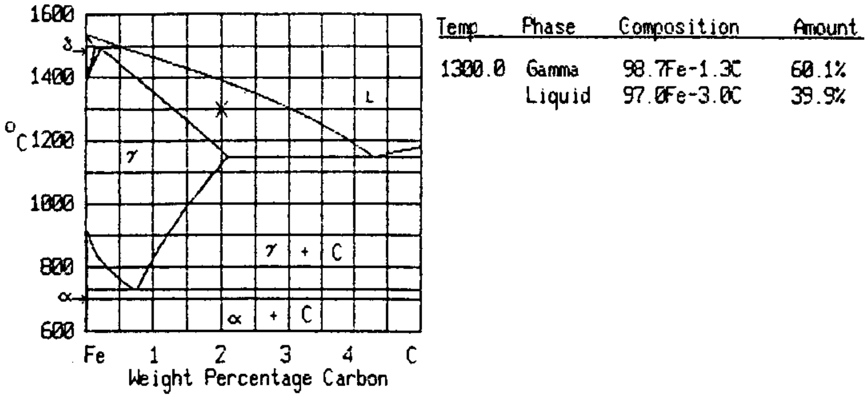
Objective: To assist students in understanding phase diagrams. Emphasis is placed on the use of the lever law, and interpretation of the phase diagrams.

Equipment: IBM compatible computer which runs MS-DOS or PC-DOS, with a minimum hardware configuration of 256K with CGA. Software is the "Use of Phase Diagrams", Engineering Materials Software Series, Dahl, K. S., Van Tyne, C. J., Institute of Metals, London.

Introduction: The topic of phase diagrams is never an easy subject for students to understand. Lectures, and homework assignments using the "lever law" may assist understanding, but students seem to stumble over this relatively easy topic, despite the many ways the subject is covered.

In an attempt to present this subject in lab, I have begun using a software program which illustrates various phase diagram on the computer screen and uses the lever law to calculate weight fractions of a phase. This same program is later used in a junior-level course to cover the subjects of solidification, heat treatment and an in-depth study of phase diagrams.

Procedure: Students are given a write-up which instructs them on the use of the program. They begin by selecting the simple diagrams or eutectics option. There are several alloy systems listed. Once a selection is made, the user is asked if they would like a phase analysis, or fraction chart (amount vs. composition, or amount vs. temperature). When phase analysis is chosen, the phase diagram is displayed . A temperature and composition must be specified to see your location (asterisk designation) on the phase diagram. The results from use of the lever law, as well as phase compositions using a tie line are then shown.



```
Temp    Phase   Composition   Amount

1300.0  Gamma   98.7Fe-1.3C   60.1%
        Liquid  97.0Fe-3.0C   39.9%
```

Figure 1. Fe-C phase diagram with compositions and weight fractions of phases noted for a 2.0% C alloy at 1300°C.

Figure 1 illustrates the Fe-C phase diagram that is shown on the computer screen. A temperature of 1300°C and a composition of 2.0% C has been selected. As shown, the weight fraction of gamma is 60.1% and the weight fraction of liquid is 39.9% The composition of the gamma phase is 98.7 Fe - 1.3 C and the composition of the liquid is 97.0 Fe - 3.0 C.

If fraction charts are specified, a plot of the amount of each phase present as a function of either temperature or composition is given. Figure 2 illustrates a weight fraction chart for the Fe-C alloy system at a temperature of 800°C.

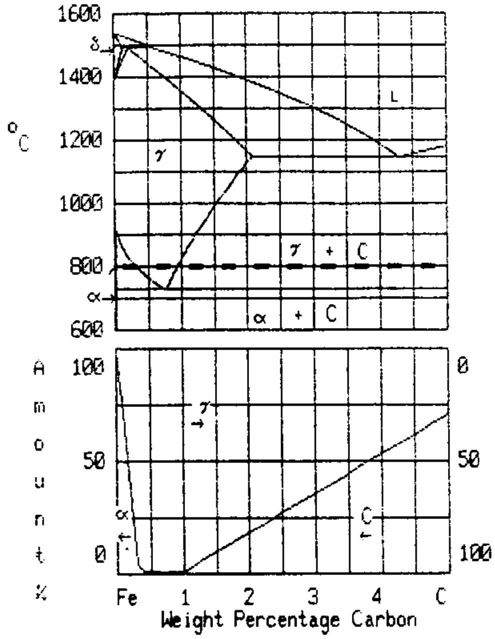


Figure 2. Weight fraction chart
for the Fe-C alloy system at 800°C.

In a junior-level materials course, solidification is studied. When the solification option is chosen, the phase diagram, cooling curve and microstructure of the solidifying material is shown on the screen. Figure 3-6 illustrate the screens that are presented for the solidification of the hypereutectic alloy.

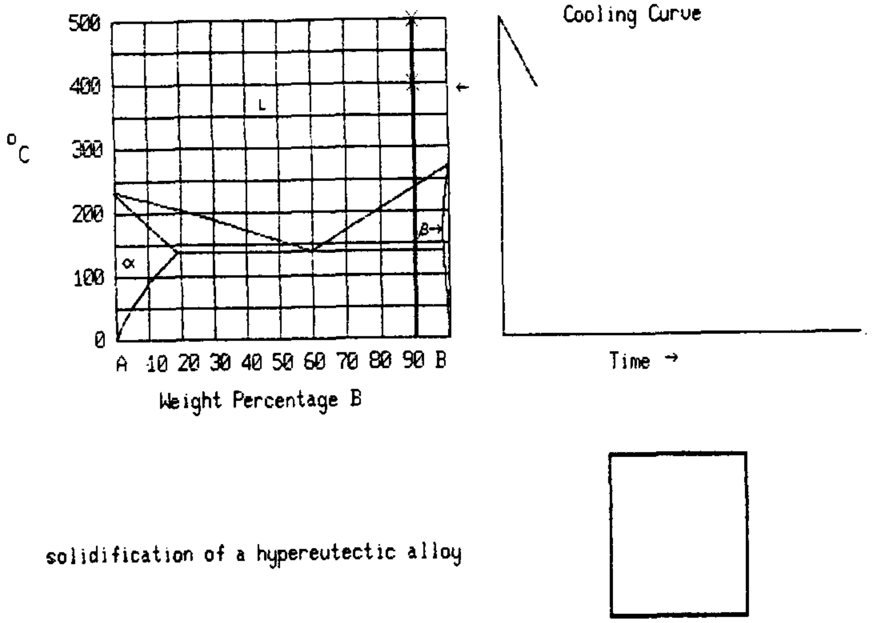


Figure 3. Solidification of a 10% B alloy. Microstructure is liquid.

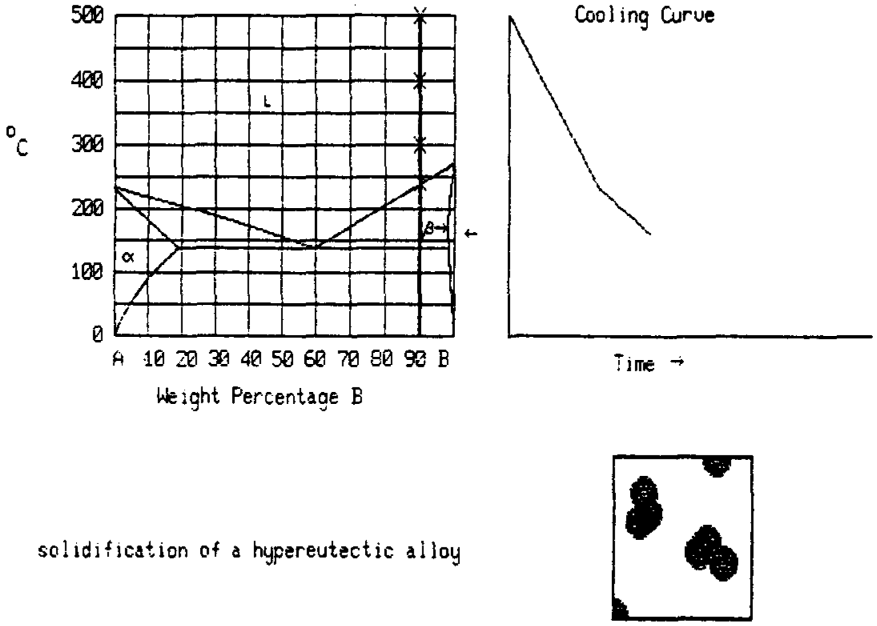


Figure 4. Solidification of a 10% B alloy. Proeutectic B is present in the microstructure.

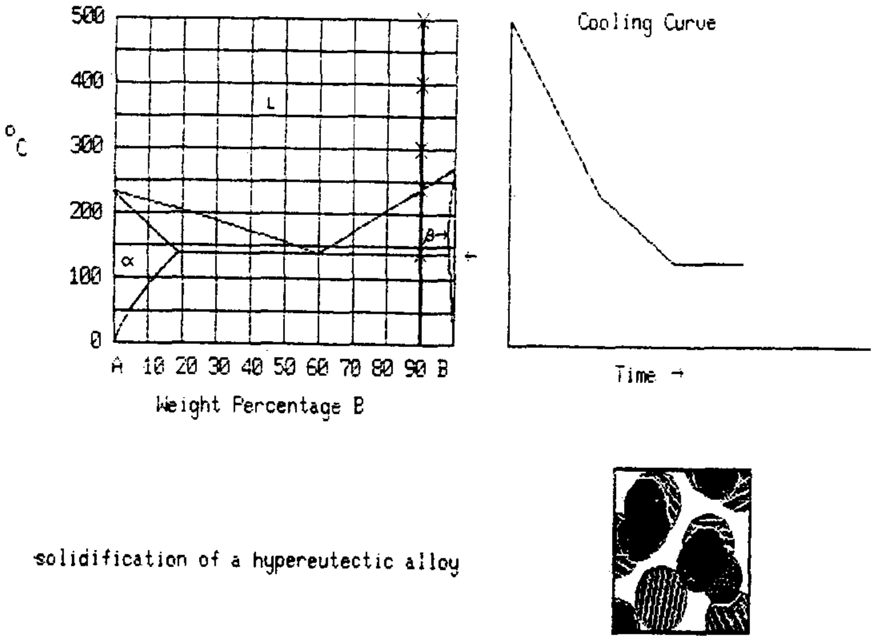


Figure 5. Solidification of a 10% B alloy. Proeutectic B is growing and the eutectic (A + B) is beginning to form.

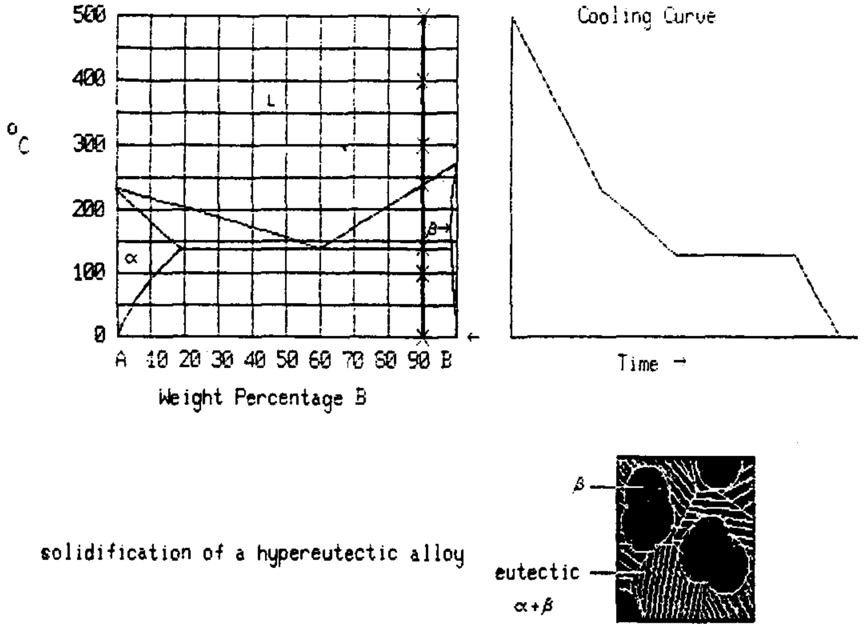


Figure 6. Solidification of a 10% B alloy. Proeutectic B and eutectic (A + B) is present in the microstructure.

Students are required to complete a table, which asks for the weight fractions and composition of 20 alloy/temperature combinations. The program will show students the correct answers, but all answers must be verified by hand using the lever rule, and tie line.

I have found that students enjoy learning from a computer (even the subject of phase diagrams). They begin to learn how to interpret phase diagrams, and understand the use of the tie line and lever rule. This program helps students to understand the difference between composition and weight fraction, and also between eutectic and eutectoid reactions. The subject of phase transformation and the interpretation of phase diagrams is a difficult subject for students to understand, but I believe this software program helps students to better grasp a difficult subject.

Note to Instructor: The lab write-up consists of a general tutorial for using the software, along with the problem assignment. This exercise is best presented in a self-taught environment, where students learn for themselves. They should be able to pace themselves depending on their computer skills, understanding of the subject, etc.. The instructor serves as a reference, but does not "teach" the subject.

References:

Dahl, K. S., van Tyne, C. J., The Use of Phase Diagrams, operating manual, Institute of Metals, London, England, 1988.

PRECISION MEASUREMENTS OF THE MICROWAVE DIELECTRIC CONSTANTS OF POLYVINYL STEARATE AND POLYVINYLIDENE FLUORIDE AS A FUNCTION OF FREQUENCY AND TEMPERATURE

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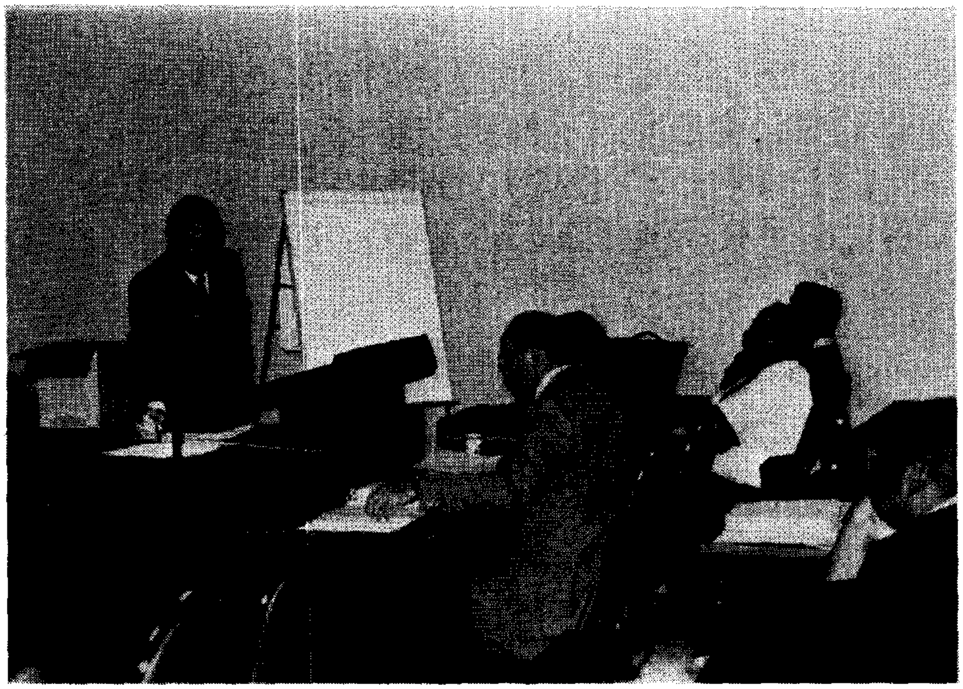

Precision Measurements of the Microwave Dielectric Constants of Polyvinyl Stearate and Polyvinylidene Fluoride as a Function of Frequency and Temperature.

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KEY WORDS

Dielectric constant, microwave resonant cavity, polymers and Dielectric relaxation.

OBJECTIVES

1. To tune a microwave resonant cavity.
2. To operate a microwave spectrometer at different frequencies.
3. To operate thermal baths for heating and cooling the resonant cavity.
4. To study dielectric relaxation and analyze data for dielectric constant measurement.

SUMMARY

A microwave spectrometer in the x-band of frequencies is designed to study dielectric behavior of polar and non-polar molecules. The resonant cavity in the spectrometer is used as a probe to determine the complex permittivity of a sample of polyvinyl stearate and polyvinylidene fluoride. A very sensitive heating and cooling technique is designed to study the microwave response of these materials at different temperatures. The real and imaginary parts of the complex permittivity are calculated by using Slater's perturbation equations and Debye's equations are used to calculate the relaxation times.

INTRODUCTION

Polymers are large molecules of nonmetallic elements composed of many repetitive units called mers just as crystals have many unit cells. The polymer molecules are very large in size and because of this they are often known as macromolecules. The molecular sizes of the polymers can be defined in terms of the degree of polymerization which is the ratio of the molecular weight of the molecule to its mer's weight. Polyvinyl stearate represents a number of groups of polymers like polyacrylates and polyvinyl esters. The polyvinyls are linear molecules. These molecules possess a chain of carbon atoms that are covalently bonded. Vinylidene polymers are produced when two of the hydrogen atoms in ethylene are being substituted by a group of atoms. When a stress is applied to such material, the larger atoms, such as chlorine or fluorine interfere with the sliding of the chains and this process increases the strength of the polymer.

The present experiment involves the use of a microwave resonant cavity in the TE_{011} mode to study the dielectric response of polyvinyl stearate and polyvinylidene fluoride at microwave frequencies of 9.2 and 9.7 GHz. The dielectric relaxation of these polymers is also studied as a function of temperature. It has proven to be a very interesting experiment to find the dielectric response of a material near a phase transition region i.e., going from an ordered to a disordered phase. It was shown by Smyth (Ref. 1) that from measurements of the static dielectric constant of compounds as a function of the temperature, important conclusions concerning molecular freedom may be drawn. A number of experiments have been done to study the dielectric behavior of a number of liquids and solids (2-7). The dielectric response of a polar liquid shows a large change at the freezing point. A typical example is that of nitromethane as studied by Smyth and Walls (Ref. 8). The measurements of this polar substance were carried out at a frequency of about 7×10^4 Hz. The drop in the dielectric constant of nitromethane from 45.5 to 3.93 at the freezing point is caused by the partial or complete loss of rotational polarization (freedom to reorient), while the electronic and vibrational polarization remain relatively unchanged. Smyth and Hitchcock (Ref. 9) made similar measurements on nitrobenzene and found that the dipole relaxation occurred at rather large frequencies. On the basis of the x-ray studies, indications of side-chain crystallinity have been reported for polyvinyl esters and polyacrylates (Ref. 10 and 11). The room temperature values of the dielectric constant and loss tangent for the polyethylene are 2.25 and less than 0.0002 respectively as shown by Von Hippel (Ref. 12).

THEORY

In his theory of polar molecules, Debye (Ref. 13) explained the relaxation phenomenon and showed that polarization of a dielectric medium in an electric field might arise from the partial orientation of permanent molecular dipoles by the field as well as from the distortion of electronic orbits in the molecules. The theory starts from an assumption about the time variation of the polarization in response to a step function removal of the field. The simplest assumption is of an exponential decay:

$$P(t) = P_o\, e^{-t/\tau} \tag{1}$$

where P_0 is the polarization of the medium in the presence of an applied field in the form of a step function, P(t) is the time response of the polarization when the applied field is removed and τ is the relaxation time.

Further, using his simple model, Debye explained the process of molecular relaxation by assuming the dipolar molecule to be a sphere of radius a moving in a continuous viscous fluid possessing a coefficient of internal friction η and obtained an expression for the relaxation time

$$\tau = \frac{4\pi a^3}{kT}\eta \tag{2}$$

where k is the Boltzmann's constant, T is the absolute temperature, a is the radius of the Debye sphere and η is the viscosity of the medium.

The above equation is valid for systems in which there is no interaction between dipoles. This requires dilute solutions of dipolar molecules in a non-polar liquid.

The fact that the relaxation time depends so strongly on the temperature indicates that at some stage in the relaxation process a particle needs a certain amount of energy in excess of the average thermal energy to overcome a potential barrier. Thus, the exponential temperature dependence of τ and η is similar to that of chemical reaction rates where we also deal with the case that a particle has to acquire a certain activation free energy. This possibility of approaching the dielectric relaxation from the standpoint of chemical rate theory was first pointed out by Eyring (Ref. 14) and developed by Kauzmann (Ref. 15).

The dielectric properties of different materials can be expressed in terms of the complex permeability μ^*, the complex permittivity ϵ^*, and their relative loss tangents. These are expressed as follows.

$$\mu^* = \mu' - j\mu'' \tag{3}$$

with

$$\tan\alpha = \frac{\mu''}{\mu'} \tag{4}$$

where μ' and μ'' are the real and imaginary parts of the complex permeability.

and

$$\epsilon^* = \epsilon' - j\epsilon'' \tag{5}$$

with

$$\tan\delta = \frac{\epsilon''}{\epsilon'} \tag{6}$$

where ϵ' and ϵ'' are the real and imaginary parts of the complex permittivity.

The polarization of a certain material can be measured in terms of these quantities (μ^* and ϵ^*). Dispersion is shown in a number of materials with variation of the complex permeability and the complex permittivity. There are basically two different kinds of dispersions shown in materials. In the first type of dispersion the polarizations are unable to follow the fields at higher frequencies. This is known as the Debye relaxation. The second type of dispersion is associated with quantized transitions taking place from one energy level to another in the atom or molecule.

According to Slater's perturbation theory for resonant cavities, the phase shift and the cavity Q changes are functions of the dielectric properties and its perturbation change (Ref. 16). Only the final results are given here and are:

$$\frac{\Delta\omega}{\omega} = \left(\frac{\epsilon'-1}{2}\right) \frac{\int \vec{E}_s\cdot\vec{E}\,dv}{\int \vec{E}\cdot\vec{E}_a dV} \tag{7}$$

and

$$\Delta\left(\frac{1}{Q}\right) = \epsilon'' \frac{\int \vec{E}_s\cdot\vec{E}\,dv}{\int \vec{E}\cdot\vec{E}_a dV} \tag{8}$$

ω is the angular frequency $2\pi f$, where f is in Hertz. $\Delta\omega/\omega$ is the phase shift, E represents the electric field of the perturbed cavity, E_a is the electric field of the unperturbed cavity, E_s is the local field of the sample, and v and V are the volumes of the sample and the cavity, respectively.

EXPERIMENTAL PROCEDURE AND RESULTS

A microwave resonant cavity in the TE_{011} mode was used as a probe in a microwave spectrometer in the x-band of frequencies. The details of this spectrometer are given elsewhere (Ref. 2) but for convenience a block diagram is shown in Figure 1 with major components. A 2k25 klystron is used in this experiment and this is slightly tunable around 9.0 GHz. The dielectric behavior of polyvinyl stearate was studied at 9.2 GHz as a function of temperature and that of polyvinylidene fluoride was studied as a function of frequency between 8.8 and 9.7 GHz. Figure 2 shows the schematic for a very sensitive thermal bath for heating and cooling the cavity to get desired temperature for dielectric relaxaton studies.

The shift in ω and change in Q are dependent upon the location as well as the dielectric nature of the perturbing sample. In this experiment the perturbing sample was placed along the symmetry axis of the cylindrical cavity. The resultant change in ω and change in Q were monitored as the cavity temperature was varied. The two parameters, ω and Q are related to the real and imaginary parts of the complex pemittivity (ϵ^*) of the perturbing sample and measurements on these two parameters enable the values for ϵ' and ϵ'' to be obtained via Equations 7 and 8 respectively.

Figures 3 and 4 show the behavior of frequency shift and Q-change as a function of temperature. From Slater's equations (#7 and 8) one can see that the frequency shifts are related to ϵ' the real part of the dielectric constant and the Q-changes on the other hand are related to ϵ'', the imaginary part of the complex dielectric constant (ϵ^*). As can be seen from these figures that there is a dramatic change in the dielectric behavior of polyvinyl stearate near its phase transition temperature of about 51.6°C. The real and imaginary parts of the complex permittivity change sharply around that temperature. The real part of the dielectric constant (ϵ') remains constant after the phase transition temperature but the imaginary part (ϵ'') shows a uniform decrease as the temperature is changed up to around 100°C.

Figures 5 and 6 display the dielectric behavior for a sample of polyvinylidene fluoride as a function of frequency. The frequency shift that corresponds to the real part of the dielectric constant shows higher values at lower frequencies. Right around 9.3 GHz there seems to be a discontinuity in the value of the dielectric constant and after that it decreases up to a frequency of about 9.6 GHz and then levels off. The dielectric loss term (ϵ'') shows an increase as the frequency increases up to around 9.2 GHz and then its value decreases as the frequency increases.

Microwave resonant cavity used in this experiment was very successful in monitoring the phase changes in polyvinyl stearate and polyvinylidene fluoride. The dielectric behavior of these two polymers was studied very precisely with this technique. The thermal bath designed to take the data at various temperature was very sensitive. With this technique the temperature of the sample could be maintained at a desired value to study the dielectric behavior more precisely at that value. It is very critical to maintain the temperature of the substance under study as it goes through its phase transition temperature. In this experiment this could be achieved very successfully.

REFERENCES

1. Smyth, C. P.: Dielectric Polarization of Liquids V. Atomic Polarization. J. Am. Chem. Soc., 51, 1929, p. 2051.

2. Dahiya, J. N., Jani, S. K., and Roberts, J. A.: Phase Transition Studies in Polar and Non-Polar Liquids at Microwave Frequencies. J. Chem. Phys, 74(6), 1981, p. 3609.

3. Benedict, T. S., and Durand, J. L.: Dielectric Properties of Single Domain Crystals of BaTiO3 at Microwave Frequencies. Phys. Rev. 109, 1958, p. 1091.

4. Birnbaum, G., and Franeau, J.: Measurements of the Dielectric and Loss of Solids and Liquids by a Cavity Perturbation Method. J. Appl. Phys., 20, 1949, p. 817.

5. Collie, C. H., Hasted, J. B., and Ritson, D.M.: The Dielectric Properties of Water and Heavy Water. Proc. Phys. Soc., 60, 1948, p. 145.

6. Crouch, G. E.: Dielectric Measurement of Liquids at Microwave Frequencies. J. Chem. Phys., 16, 1948, p. 364.

7. Gevers, M.: Measuring the Dielectric Constant and the Loss Angle of Solids at 3,000 Mc/s. Philips Tech. Rev., 13, 1951, p. 61.

8. Smyth, C. P., and Walls, W. S.: Dielectric Investigations on Nitromethane and Chloropicrin. J. Chem. Phys., 3, 1935, p. 557.

9. Smyth, C. P., and Hitchcock, C. S.: Dipole Rotation in Crystalline Solids. J. Am. Chem. Soc., 54, 1932, p. 4631.

10. Fitzgerald, Edwin R.: Mechanical Resonance Dispersion in Crystalline Polymers at Audio-Frequencies. J. Chem. Phys., 27, 1957, p. 1180.

11. Fitzgerald, Edwin R.: Dynamic Mechanical Properties of Polyvinyl Stearate at Audio-Frequencies. J. Appl. Phys., 29, 1958, p. 1442.

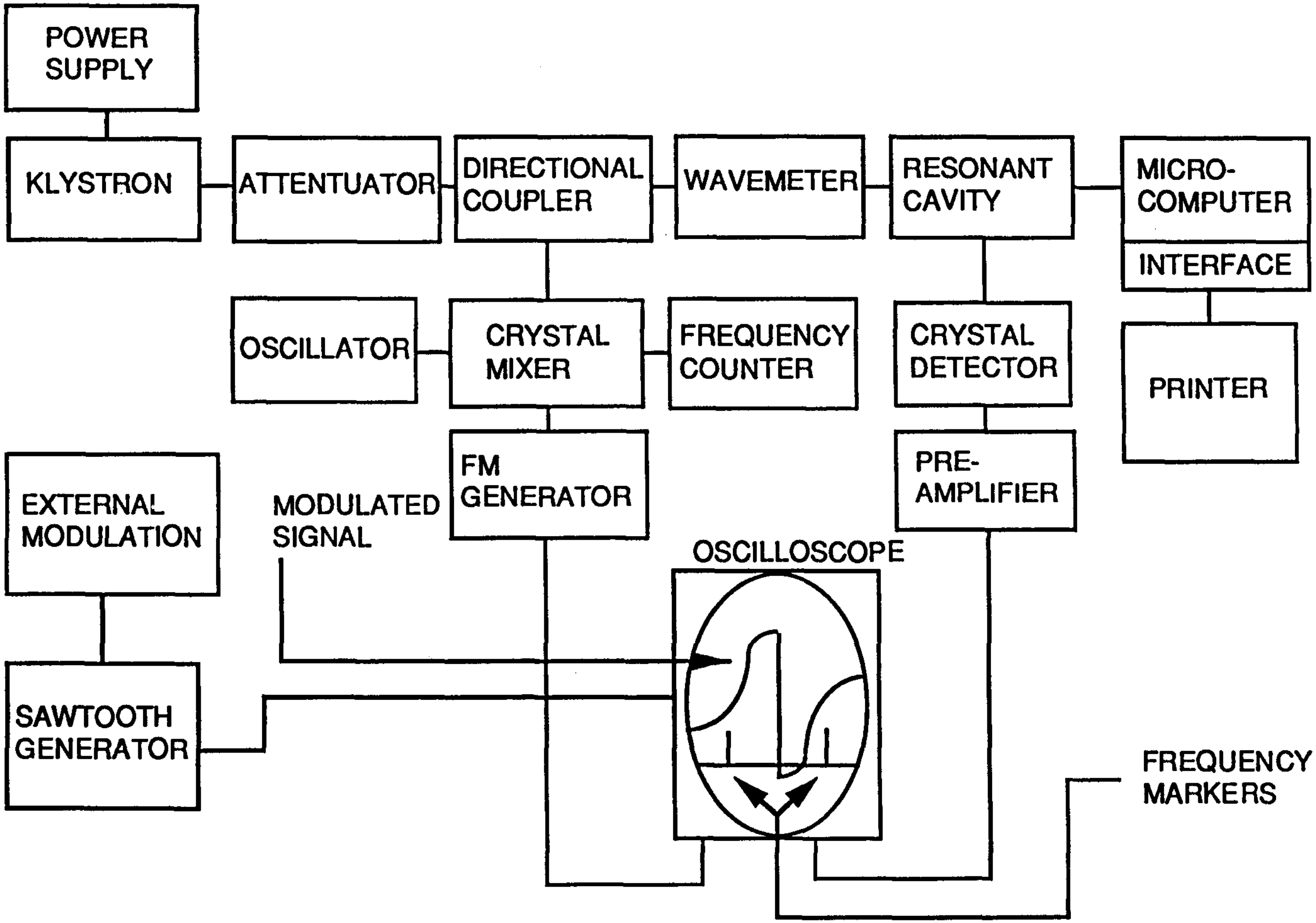
12. Von Hippel, A. R.: Editor, Technology Press of M.I.T. and J. Wiley and Sons, N.Y., 1954.

13. Debye, P.: Polar Molecules. Chemical Catalog, New York, 1929, Chap. V.

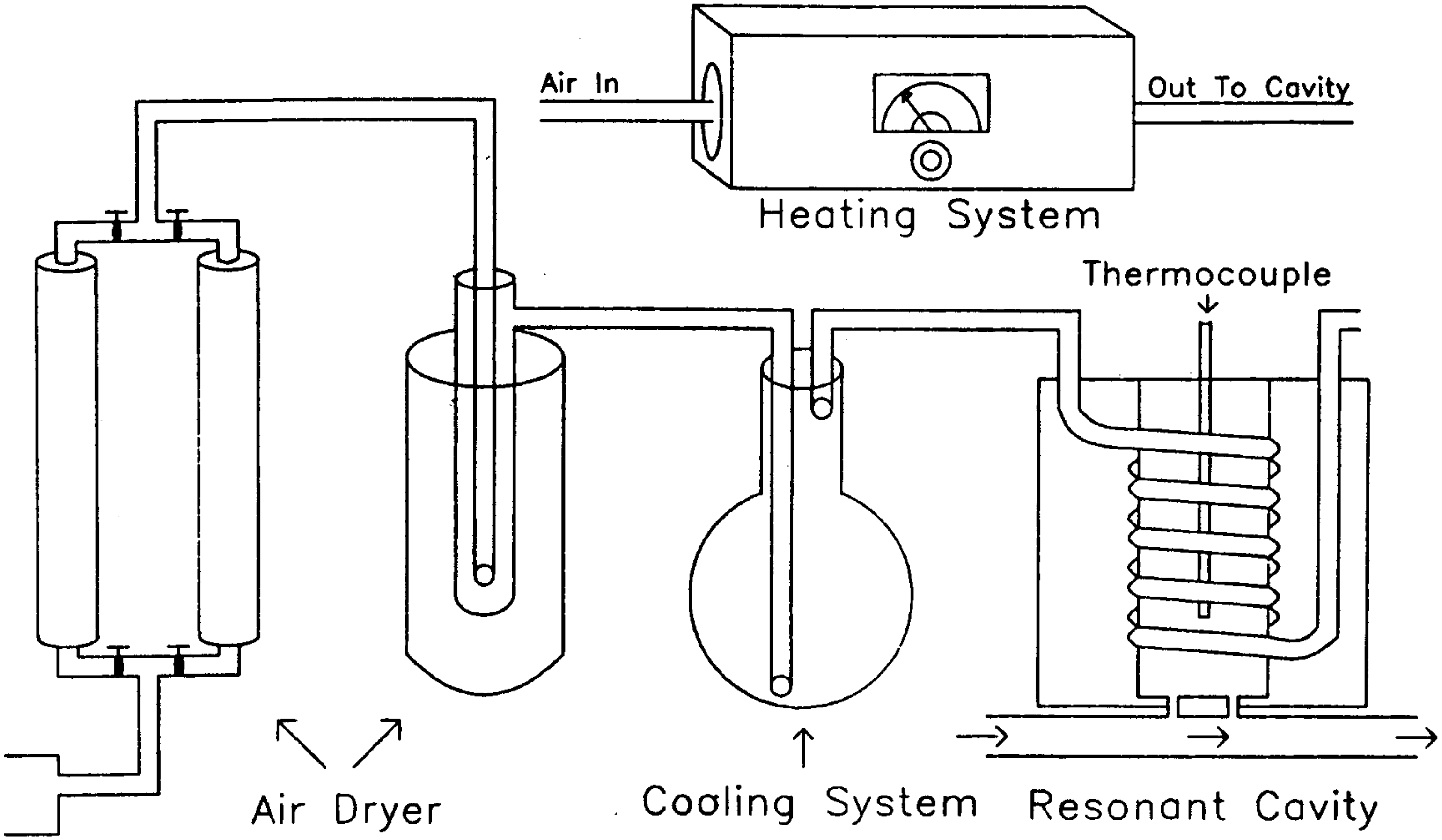
14. Stearn, Allen E., and Eyring, Henry: The Deduction of Reaction mechanism from the Theory of Absolute Rates. J. Chem. Phys., 5, 1937, p. 113.

15. Kauzmann, Walter: Dielectric Relaxation as a Chemical Rate Process. Rev. Mod. Phys., 14, 1942, p. 12.

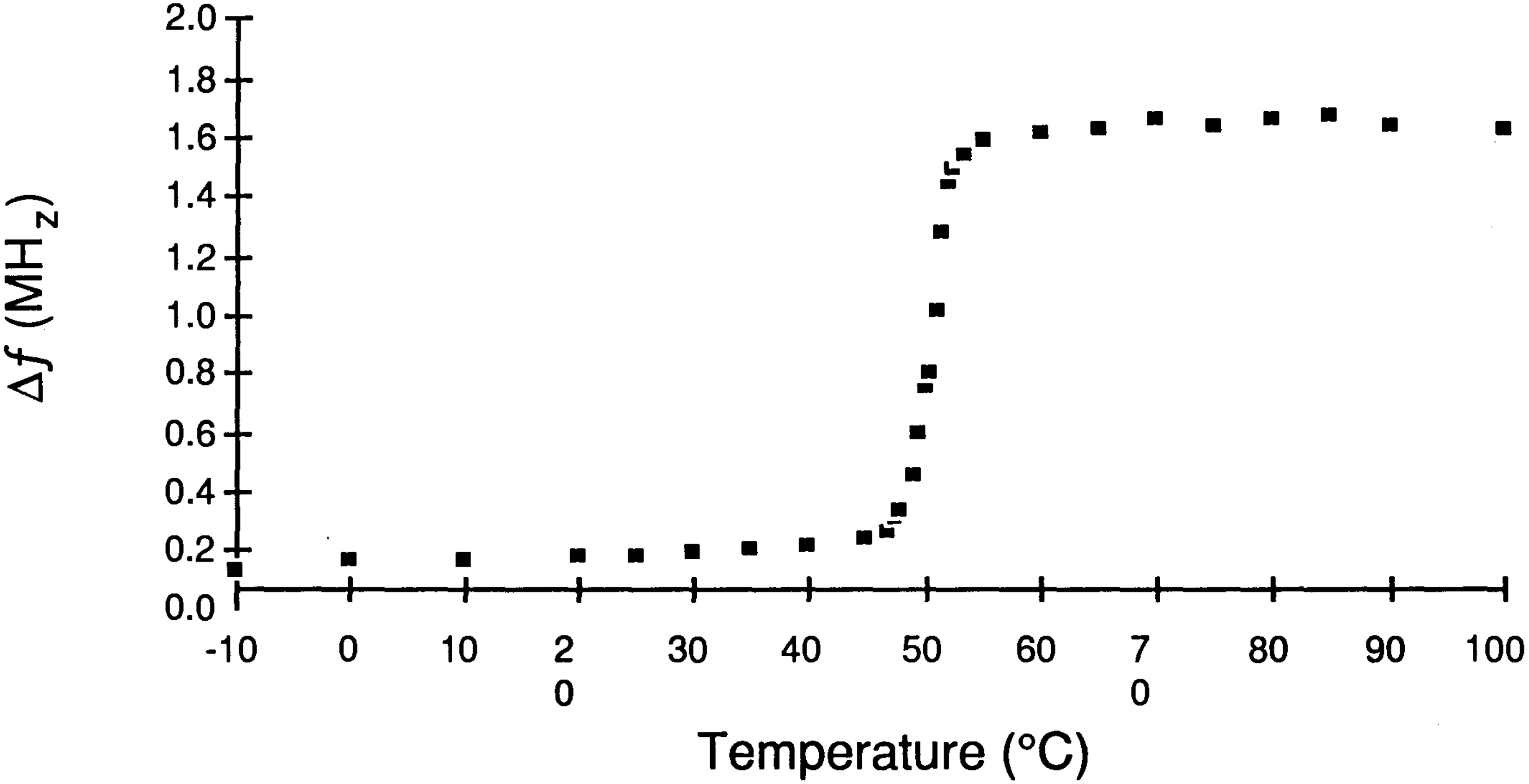
16. Slater, J. C.: Perturbation Theory for Resonant Cavities. Rev. Mod. Phys., 18, 1946, p. 441.



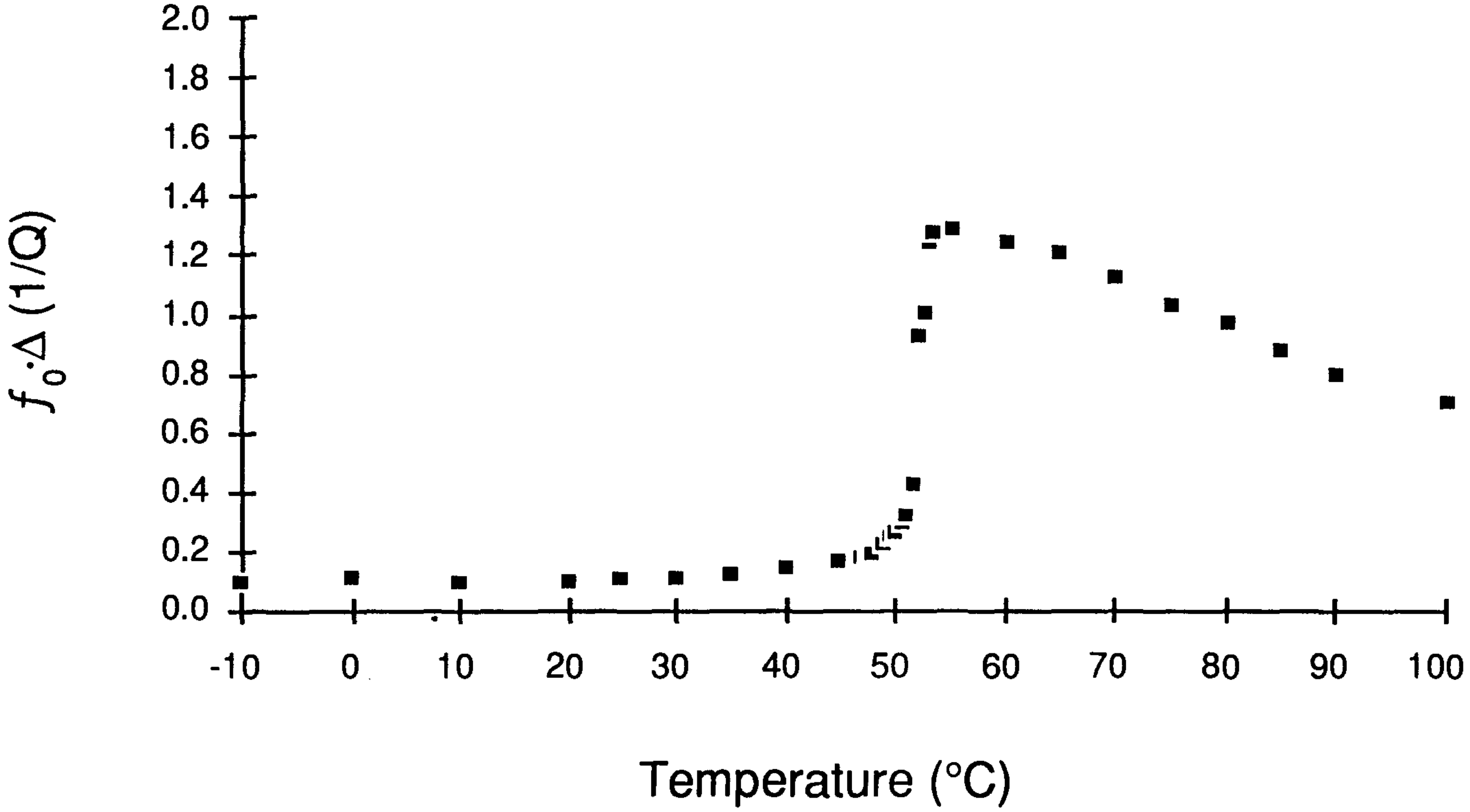
1. Block diagram of the microwave spectrometer used in this experiment.



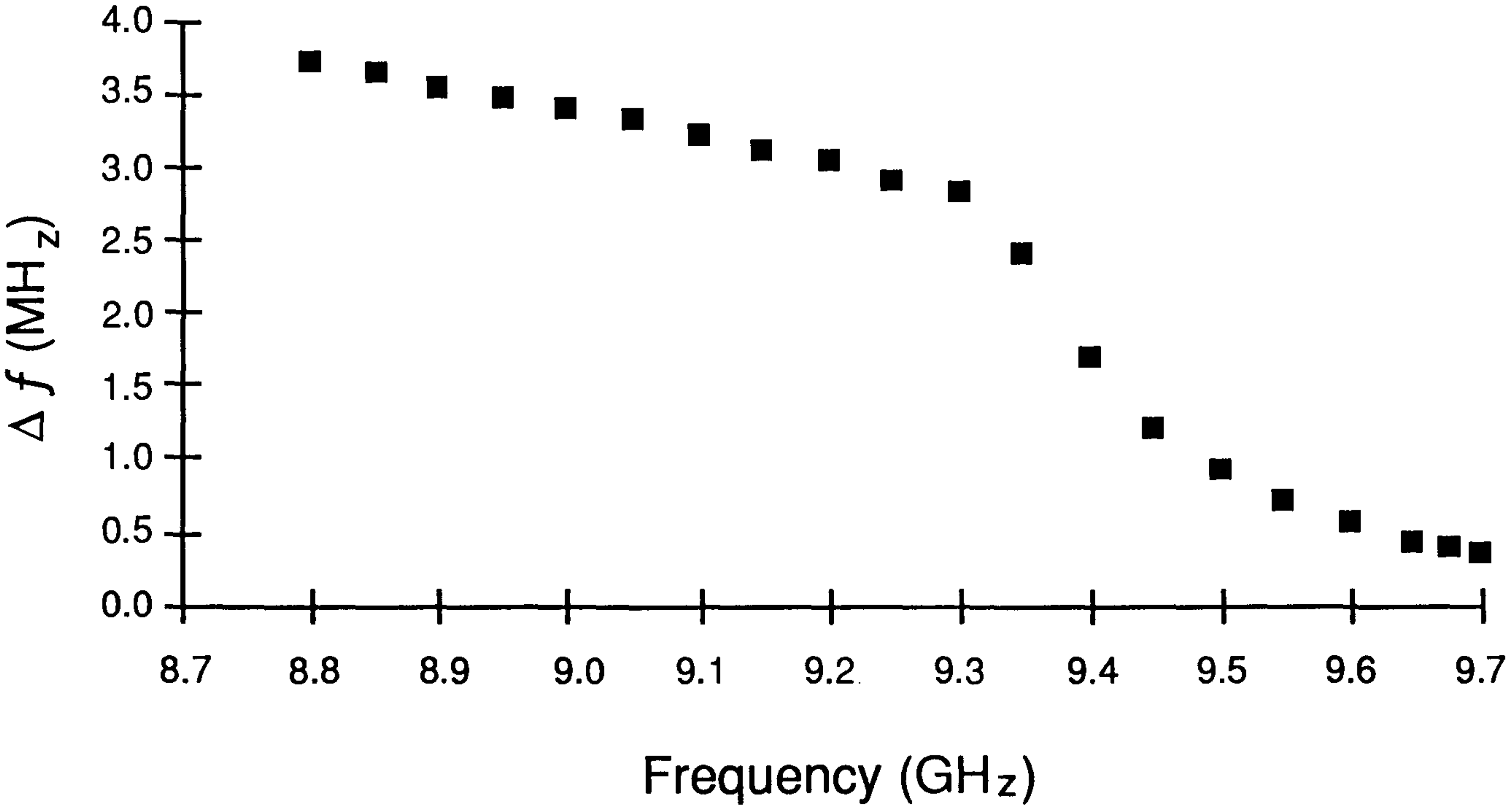
2. Block diagram of the heating and cooling system used in this experiment.



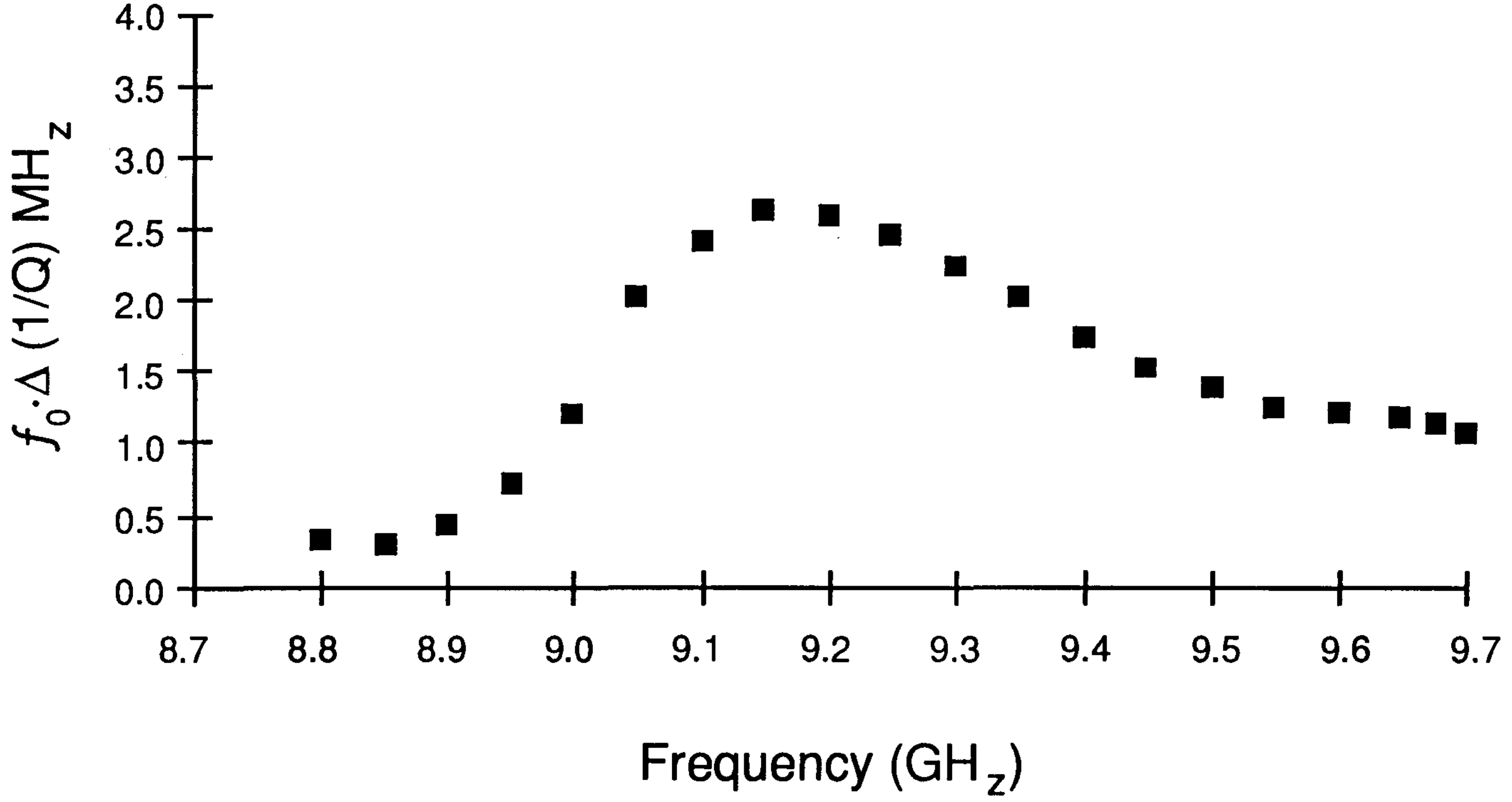
3. Frequency shift (Δf) as a function of temperature for a sample of polyvinyl stearate.



4. Quantity $f_0 . \Delta(1/Q)$ as a function of temperature for a sample of polyvinyl stearate.



5. Frequency shift (Δf) as a function of the microwave resonant frequency for a sample of polyvinylidene fluoride.



6. Quantity f_0. $\Delta(1/Q)$ as a function of the microwave resonant frequency for a sample of polyvinylidene fluoride.

EXPLORING THE CRYSTAL STRUCTURE OF METALS

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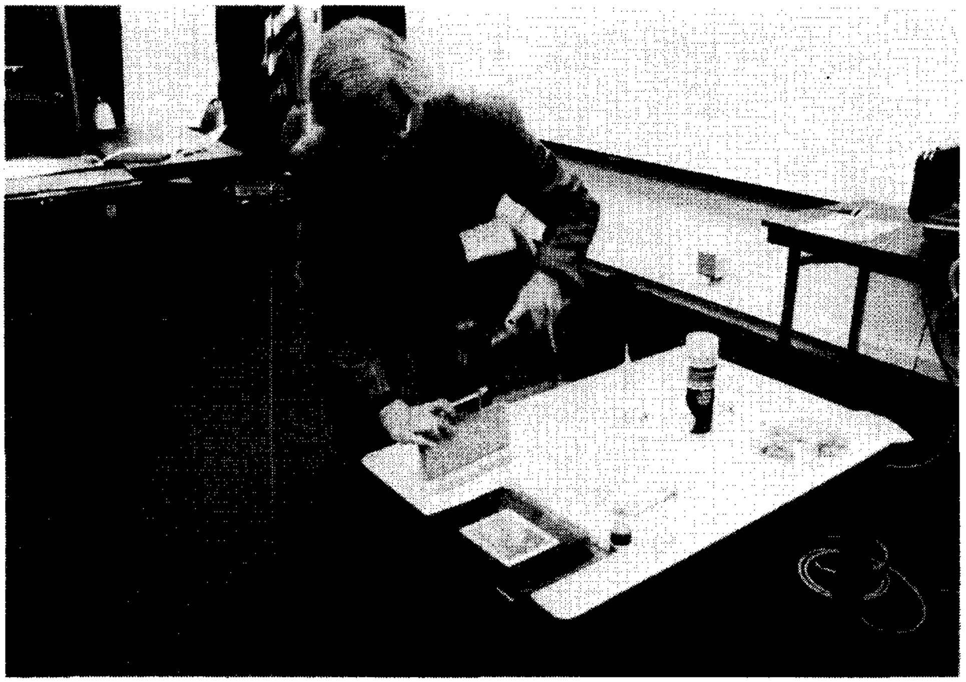

EXPLORING THE CRYSTAL STRUCTURE OF METALS

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KEY WORDS: Crystal Structure, Cleavage, Plastic Deformation, Alloys, Glassy (Amorphous) Structure

PREREQUISITE KNOWLEDGE: Introduction to crystal structure and atomic bonding, as covered in a middle school or junior high school earth science class.

OBJECTIVE: These activities are intended to supplement the study of mineral crystal structure in a middle school earth science class. The simple metallic close-packed structures are explored through model building, and differences in structure are related to mechanical behavior. The concepts of alloying and glassy (amorphous) stucture are introduced, and give students a glimpse of what a materials scientist does. Some of these approaches may prove useful in a college-level introductory materials science class.

EQUIPMENT/MATERIALS:
1. Magnetic Marbles (120 required for student activity and permanent models)
2. Spray Craft Glaze
3. Zinc, Zinc-Aluminum, and Aluminum Rods, and Jig for Holding Both Ends of Rod for Impact Test.
4. Galvanized steel with distinctive surface grains or "spangles".
5. Hammer

INTRODUCTION

Crystal structure generally holds the fascination of students at the middle school/junior high level, because of the inherent beauty of faceted mineral crystals. Standard curriculum typically includes identification of mineral samples, introduction to the various crystal lattice symmetries, and discussion of the kinds of atomic bonds. Metallic structures receive minimal attention.

Using magnetic marbles, students are challenged to construct the two close packed metallic crystal structures. These activities can be structured as team races to stimulate interest. Differences in structure can be related to deformation behavior, and this relationship can be demonstrated by a simple impact test.

MAGNETIC BALL MODELS OF CLOSE-PACKED METAL STRUCTURES

The simplified picture of the metallic bond is that of core atoms packed in a "sea" of disassociated valence electrons. This allows construction of models of the two close-packed structures using magnetic marbles. (Students should be reminded that the forces holding the atoms together are not the same as the magnetic attraction holding the marbles together.)

A good in-class approach is to have two groups of four students participate in a race to build a closest-packed structure (boys vs. girls works well). Each student is given seven marbles. The first task is to make a layer of the seven "atoms" that is as tightly packed as possible. Most students can accomplish this fairly quickly. The easiest way is to make a row of six marbles, close it into a circle, and then place the seventh marble in the center. The real challenge is to stack these layers to make a three dimensional structure that is also as tightly packed as possible. Students often start with a structure having the center atom of each layer aligned. This causes the center atom of the top layer to stick out, and is not a closest packed structure. Closest packing can be attained by putting the second layer above the B or C hole sites in the first layer, and similarly nestling subsequent layers in the holes of the layer below it (Figure 1).

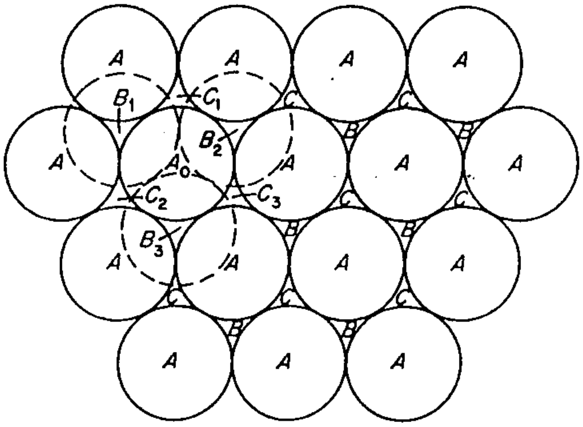

FIGURE 1
Stacking Sequences in Close-Packed Crystal Structures
(from R.E. Reed-Hill, Physical Metallurgy Principles, Van Nostrand Reinhold Co., New York, 1964)

Once the students' race is over, the instructor can explore details of the structures. It is helpful to "cheat" by building models with layers that have been glued together using spray craft glaze. There are two basic options in stacking, the ABAB sequence of the hexagonal close-packed structure, and the ABCA of the face-centered cubic structure. (I do not mention face-centered cubic to younger students, as it tends to confuse them; instructors should be able to point out the faces of the cubic unit cell revealed by the model).

By adding individual marbles to the ABCA stacking arrangement, students can discover a key aspect of this structure: diagonal planes have the same close-packed symmetry as the base plane. On the other hand, the ABAB stacking arrangement has close packed layers only on the horiontal plane of the model. This subtle difference in structure makes a tremendous difference in mechanical behavior.

RELATION OF CRYSTAL STRUCTURE TO MECHANICAL BEHAVIOR

Assembly of the ABAB and ABCA models reveals an important relationship between metallic crystal structure and mechanical behavior. The ABCA model displays diagonal faces that have the same tightly packed arrangement as the base. This structure is associated with metals like aluminum and copper, which can be easily bent. For metals to "plastically deform", atoms have to slide over each other. This occurs more easily on these tightly packed planes. Aluminum deforms easily because it has many of these planes in different orientations. On the other hand, crystals of zinc and magnesium have the ABAB structure, and atoms slide easily only on the base layer. Zinc tends to break or "cleave" along crystal faces when it is struck with too much energy. (I generally avoid the concept of dislocations when presenting this material at the pre-college level).

To reinforce this concept, a simple impact test is performed using rods of aluminum, zinc, and zinc-5 wt.% aluminum alloy. The rods are supported at both ends in a jig, and impacted with a hammer. (Students should wear eye protection to guard against flying pieces of metal). The aluminum bar will bend, while the zinc will break by cleavage, revealing the crystal structure inside the metal. Students should note that the zinc is not one big crystal, but a number of little crystals or "grains". The grain structure of zinc can also be seen in a sample of galvanized steel, which has a coating of zinc deposited on it for corrosion protection.

If zinc and aluminum have such different properties, what will be the behavior of a blend of the two? Such a blend is called an "alloy". Hammer impact of the rod of zinc with 5% (by weight) aluminum will reveal its strength and toughness. At high magnification, the internal structure of this alloy is revealed as a fine assembly of zinc-rich and aluminum-rich regions, each with its characteristic crystal structure (Figure 2). The instructor should explain how a microscopic image like Figure 2 is obtained: a small piece of the metal is mounted in plastic, polished to a mirror finish, and then etched with an acid. The alloying of a metal to improve its properties is a good example of what a materials scientist does. By understanding the structure of a material and how it controls properties, we can design new materials that are stronger, tougher, lighter, more durable, more reliable, less costly, and better for the environment.

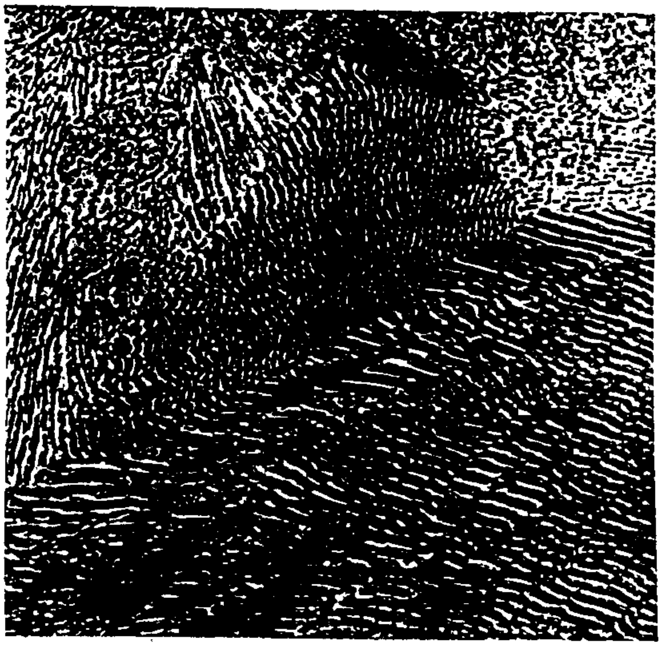

FIGURE 2
Microstructure of Zinc-5wt.% Aluminum Alloy; 1000X

GLASSY OR AMORPHOUS STRUCTURE

A final game with the magnetic marbles can be used to show the difference between crystalline and glassy (amorphous) structure. We have seen that crystals have an orderly arrangement of atoms. Nature makes these structures because they have a low energy associated with them. The other alternative is the structure of a glass, which has no long range order in the packing of the atoms. This occurs because different atoms do not fit together well, or are cooled from the liquid or vapor state so quickly that there is no time for the atoms to arrange themselves into crystals.

Two student volunteers are picked for a race to build a glassy structure using the magnetic marbles. As an incentive, the winner gets to keep the marbles. However, the loser has to perform a horrible homework assignment. This may decrease the pool of volunteers, but usually there are a few students game enough to try. The fact is that the race is fixed so that both students win. As soon as the race starts it is over, because any random cluster of marbles will have an amorphous structure. This reinforces the notion that rapid cooling from the liquid state can "freeze in" the glassy structure. Obsidian is a good mineral example of rapidly cooled glass. Snowflake obsidian shows crystals of quartz starting to form in the glassy matrix. Window glass is another example - in this case the complex chemistry of the silicate glass gives an amorphous structure even if it is cooled fairly slowly. Actually, many metallic alloys can be amorphous if they are quenched from the liquid state quickly enough (on the order of a million degrees centigrade per second!) Amorphous metals can have unusual and advantageous properties, and provide another example of the work of the materials scientist.

NOTES TO THE INSTRUCTOR:

1. Magnetic marbles are available from Edmund Scientific Co., (609) 573-6250.

2. Zinc, aluminum, and zinc-aluminum alloy rods are available from Intermet Technology, 1306 Airport Rd., Aliquippa, PA 15001, (412)378-3781.

3. If this presentation is made by a visiting professional, it is a good idea to give every student something to keep. I like to have a pre-cracked zinc rod for every two students. Students can break these by hand to reveal the cleaved facets of the cast grain structue.

ACKNOWLEDGEMENTS

This material was developed under a National Science Foundation SBIR grant to Intermet Technology. In-class presentations were performed under the "PRISM" program coordinated by ASM-International. Zinc Corp. of America provided zinc samples and access to metallographic equipment.

FLUIDS WITH MAGNETIC PERSONALITIES

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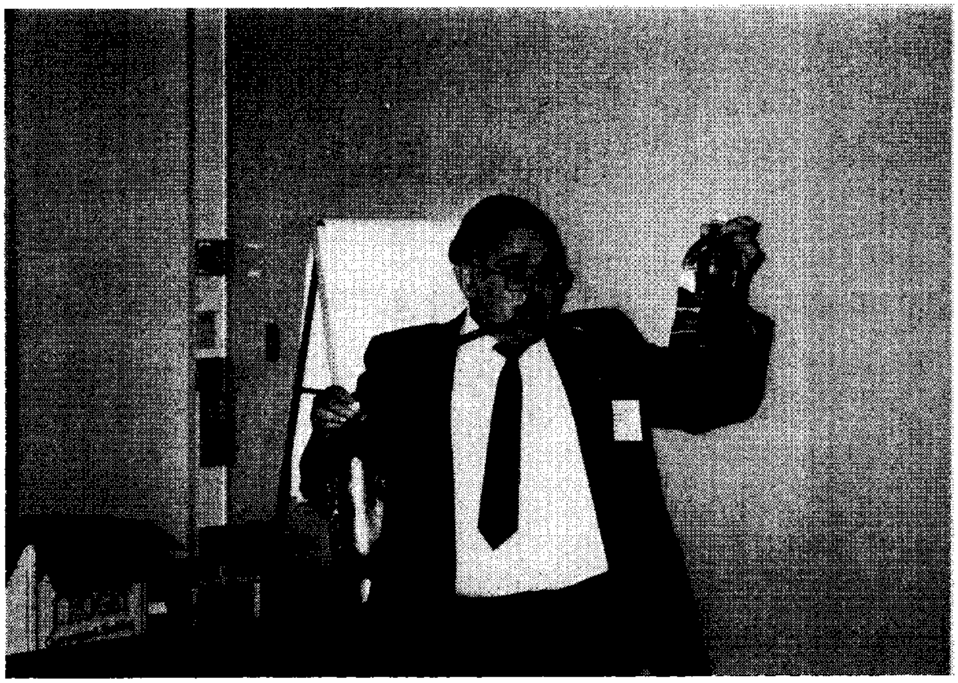

Fluids with Magnetic Personalities

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Key Words: Magnetorheological, Electrorheological, Variable Viscosity

Prerequisite Knowledge: Basic principles of electricity, magnetism, electromagnetism and viscosity.

Objective: To introduce the science of rheology. To observe the behavior characteristics of magnetorheological fluids. To demonstrate several practical applications of these fluids. To suggest some possible student laboratory experiments using magnetorheological fluids which provide data suitable for various error and statistical analyses.

Equipment:

1. Vegetable Oil (& possibly other thin oils)
2. Iron Filings
3. Various plastic bottles, tubing and Goop adhesive
4. DC power supply (2-3 amps @1-5 volts)
5. Small DC motor
6. 26-30 gauge wire
7. Several strong permanent magnets
8. Several 2 liter plastic soft drink bottles
9. Plastic Tubing, 5/16 OD, 3/16 ID, 5 feet long

Introduction to the Science of Rheology:

The term rheology means literally the study of flow. As a science, the field of rheology concerns itself with all aspects of deformation of real bodies under the influence of external stress. In traditional mechanics, the response of real bodies to external stress is studied using idealized bodies such as an ideal elastic body or an ideal viscous (Newtonian) fluid which exactly obey certain physical laws. These classical models are of practical value for classes of materials where the modeled property dominates the characteristic behavior of the material.

Real materials whose properties and behavior can be approximated by one of the classical models under one set of conditions will exhibit a different type of behavior under other properly chosen conditions. Some materials cannot be adequately described by any classical model because they exhibit properties characteristic of different models at the same time. Rheology as a science is concerned with the behavior of real materials which exhibit, to an extent that cannot be neglected, more than one fundamental property of deformation under external stress, such as elasticity and viscosity. While classical mechanics treats the solid state and the fluid state of materials separately, rheology deals with phenomena which occur in a very broad intermediate range between the solid and fluid state.

Phenomenological rheology (macrorheology) deals with real materials on a macroscopic scale and ignores the molecular nature of the material. This allows the development of mathematically continuous functions with experimentally determined parameters (coefficients) to model the observed behavior of the material[1]. The experimental apparatus presented herein can be used to provide the observational data necessary to develop and explore basic mathematically-continuous models of rheological materials.

Magnetorheological Fluids

Magnetorheological fluids are fluids which exhibit the property that their viscosity is a function of the strength of the magnetic field in which they are immersed. In a very weak magnetic field, the material behaves as a fluid. In a strong magnetic field, the material behaves as a thick slush or a weak solid. The relationship between the viscosity of the fluid and the magnetic field is continuously adjustable by varying the concentration of the magnetic material suspended in the magnetorheological fluid or by selecting a different viscosity oil as the base of the magnetorheological fluid.

Basically, a magnetorheological fluid is a suspension of magnetic particles such as iron filings in a host liquid which is typically a light oil such as light machine oil, soybean oil or other vegetable oil. When the fluid is immersed in a magnetic field, the iron filings tend to align themselves with the magnetic field and can form an elaborate solid matrix if they are plentiful enough for continuous physical contact with each other. The oil tends to adhere to the rigidly held iron filings. Obviously, the higher the concentration of the iron filings, the less likely the surrounding oil will be overcome with the gravitational tendency to flow. On the down side, the iron filings tend to be highly abrasive when in contact with other solids having relative motion to the magnetorheological fluid. While increasing the density of the iron filings in the magnetorheological fluid improves the solidification of the fluid in a magnetic field, it increases the abrasion caused by the fluid to any solid moving within the fluid.

Why Magnetorheological Materials

There are many, very broad classes of rheological materials including biological fluids such as blood, saliva, and synovial fluid; polymer melts and solutions; suspensions, emulsions and pastes. Magnetorheologic suspensions lend themselves to introductory laboratory work because they are easy and inexpensive to create, they do not require expensive or special laboratory equipment, and they adequately demonstrate rheologic behaviors.

An experiment using electrorheological fluids was presented at NEW:93[2]. Considerable applications research has been conducted using electrorheological fluids. The rheological effect is based primarily on the tendency of suspended or dissolved particles or molecules to align themselves with and remain stationary within the externally applied electric field and to restrict the movement of the solvent or suspension liquid[3]. The principle problem in working with them is the high voltage (easily 1000 V/mm) required to see the rheological behavior.

Magnetorheological fluids, which closely parallel electrorheological fluid behaviors, are generally suspensions of magnetically susceptible particles in a nonmagnetic liquid. The rheologic effect is based on the tendency of the suspended particles to align themselves and remain stationary within the externally applied magnetic field and to restrict the movement of the suspension liquid. While magnetorheologic fluids tend to be messy, they are very easy and quite safe to work with. Equipment can be as simple as vegetable oil, iron filings, and a horseshoe magnet. They satisfactorily demonstrate the rheologic behavior.

Demonstration #1: Basic Magnetorheological Effect

Fluids assume the shape of their container and tend to have flat surfaces (beyond the meniscus area). Solids, and to a lesser extent slurries, tend to retain their shape regardless of their containers. In the first demonstration, the magnetorheological fluid is observed to behave as a fluid in the absence of a magnetic field. When a sufficiently strong magnetic field is applied, the magnetorheological fluid can be shaped with a non-magnetic spatula into a form which behaves as an almost solidified slurry. When the magnetic field is removed, the fluid relaxes and assumes the shape of its container with a flat surface.

Prepare the fluid by mixing iron filings into the oil with a 4 to 1 ratio by weight. Pour the fluid into the clear plastic container as in Figure 1a. Stir with the spatula and note its behavior as a fluid. Put the magnets against the side of the container and use the spatula to form the material into a bar shaped bridge between the two magnets leaving voids on the perpendicular axis as in Figure 1b. Point out the nonfluid behavior. Remove the magnets and note that the material becomes fluid again.

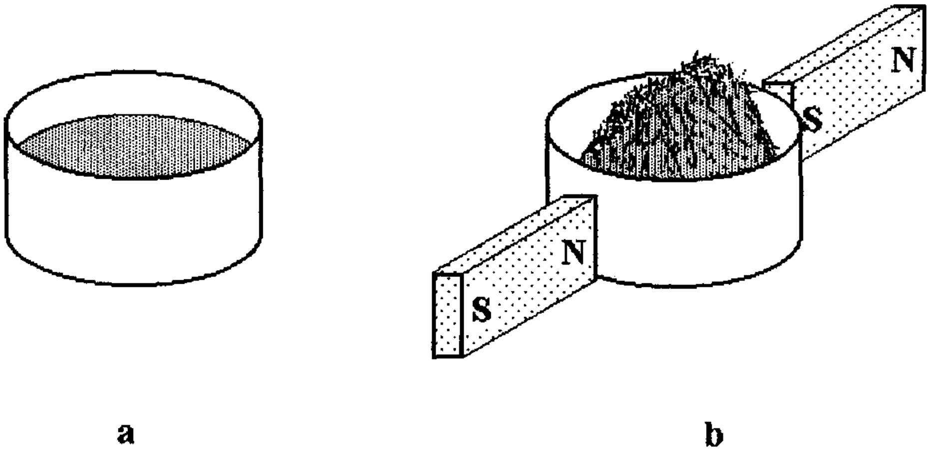

Figure 1. Basic magnetorheological effect demonstration.

Demonstration #2: Fluid Flow Rate Control

The flow rate of a magnetorheological fluid can be modulated by varying the field strength of a magnetic valve applied to a tube carrying the fluid. In this demonstration, we can vary (visibly) the flow rate of the magnetorheological fluid by varying the magnetic field strength in the flow valve.

Prepare the apparatus by drilling 5/16 in holes for the tubing into the two 2-liter soft drink bottles approximately as shown in Figure 2. Cement the two 2 foot lengths of plastic tubing in place using the Goop and allow to cure for 24 hours. The top tube in Figure 2 serves as an air return during the demonstration and as a fluid return when bottle B is inverted and raised to prepare for the next demonstration run. In a separate bottle, prepare 1 liter of the fluid by adding as much iron filing material (approximately 1/2 pound) as will stay suspended in the oil for 5 minutes. Pour the prepared fluid into bottle A and seal both bottles. The fluid can be transferred between bottles A and B by gravity. Move the fluid to bottle A and shake the bottle prior to beginning the demonstration. Raise bottle A as high as possible above B and note the flow rate of the stream into B. Apply the magnets to the lower tube as indicated in Figure 1 and note the significantly reduced flow rate. As you move the magnets closer and further away, you can continuously moderate the flow rate.

A challenging student project might be to design and build a flow rate transducer for this apparatus. A more involved student project might be to explore particle migration as the fluid flows through the tube. This effect occurs when dispersions flow through long sections of pipe[4]. Flow velocity through the tube is not uniform. Shearing stresses decrease from the surface of the tube toward the middle. It appears that the tendency for particles to migrate toward the center where shearing forces are at a minimum arise from an imbalance in the torque forces experienced by the particles in this non-uniform flow.

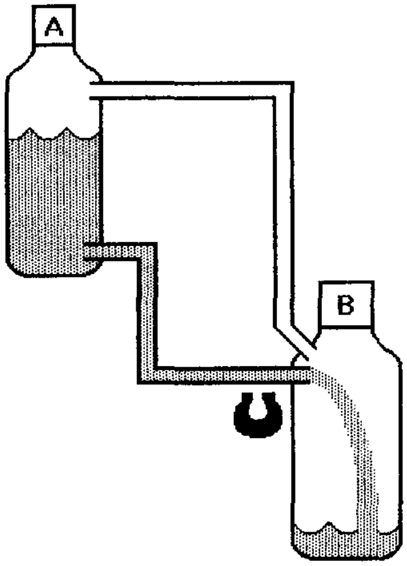

Figure 2. Flow control demonstration apparatus.

Demonstration #3: Magnetically Controlled Brake

Magnetorheological fluids can perform admirably as dynamic brakes. In this demonstration, a motor driven disk will be braked to a varying degree by varying the magnetic field applied to the magnetorheological fluid in which the rotating disk is immersed. The effect can be observed by monitoring either the rpm of the driving motor (at a fixed driving voltage) or by monitoring the current drawn by the driving motor (at a fixed driving voltage) as the magnetic field is increased.

The apparatus can be built from commonly available materials. The dimensions of the apparatus are nominal. The stirring disk is scrap aluminum cut to a circle somewhat smaller than the diameter of the fluid container. It must be center drilled to fit the threaded shaft. It should have several axial cuts creating pie shaped areas which are to be twisted to form a crude fan. The fan shape serves to help keep the iron filing suspension well mixed. The threaded shaft is brass from a toilet repair kit. The aluminum disk is mounted on the threaded shaft with two nuts by force fit. The shaft to motor coupling is a piece of insulation stripped from hook-up wire. For a more permanent situation the motor and shaft can be joined with Goop or a good epoxy cement.

A student project here might be to better instrument the apparatus by adding precise measurement of the motor's rpm. Another might be to develop a 3-D graph showing the relationship between work done, as determined by the electrical input to the system; the magnetic field strength, which could be characterized by the electrical energy supplied to electromagnets (used in place of the permanent magnets); and the density of iron filings suspended in the fluid.

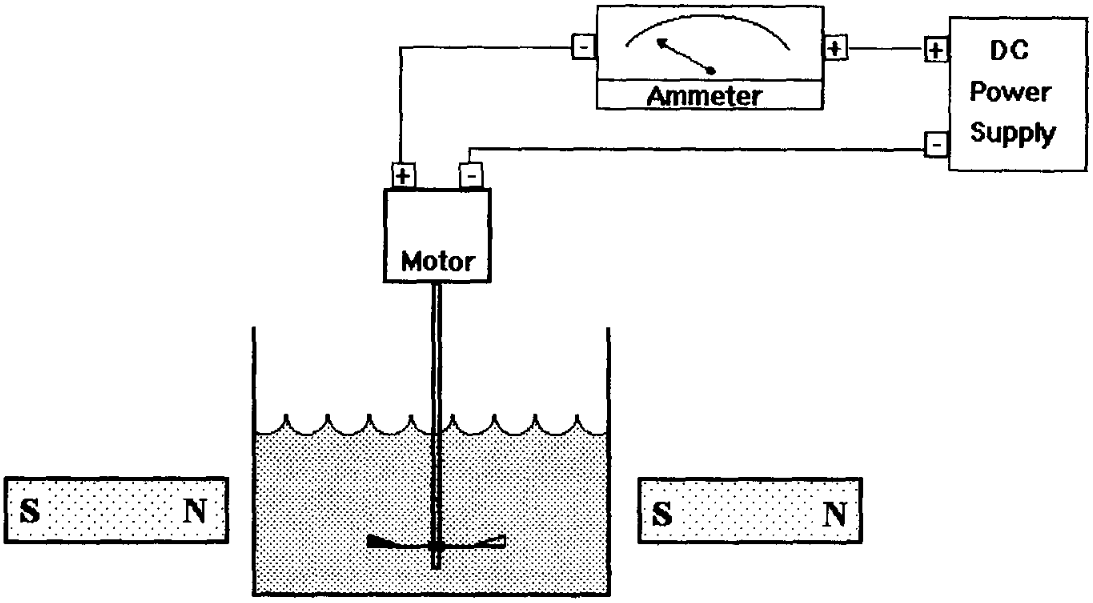

Figure 3. Magnetically controlled brake demonstration.

Notes to the Instructor:

1. Suitably small iron filings are available from Edmund Scientific as part number N60,176.
2. Magnets from Edmund Scientific as part number N40,818.
3. Goop, threaded brass rods, & plastic tubing is available in general hardware stores.
4. Suitable DC motors are from Radio Shack part nos 273-237 or 273-223.
5. Vegetable (soybean) Oil from the corner grocery store.

References:

1. Ferguson, J.; and Kemblowski, Z: *Applied Fluid Rheology*, Elsevier Science Publ. Ltd., 1991, pp 3-8.
2. Marshall, J. A.: Liquids that take only milliseconds to turn into Solids, *Prodeedings of National Educators Workshop:Update93*, pp 315-322.
3. Halsey, T. C.; and Martin, J. E.: Electrorheological Fluids, *Scientific American*, vol. 269, no. 4, Oct 1993, pp 58-64.
4. Ferguson, J.; and Kemblowski, Z: *Applied Fluid Rheology*, Elsevier Science Publ. Ltd., 1991, pp 204-205.

CDROM TECHNOLOGY TO STRENGTHEN MATERIALS EDUCATION

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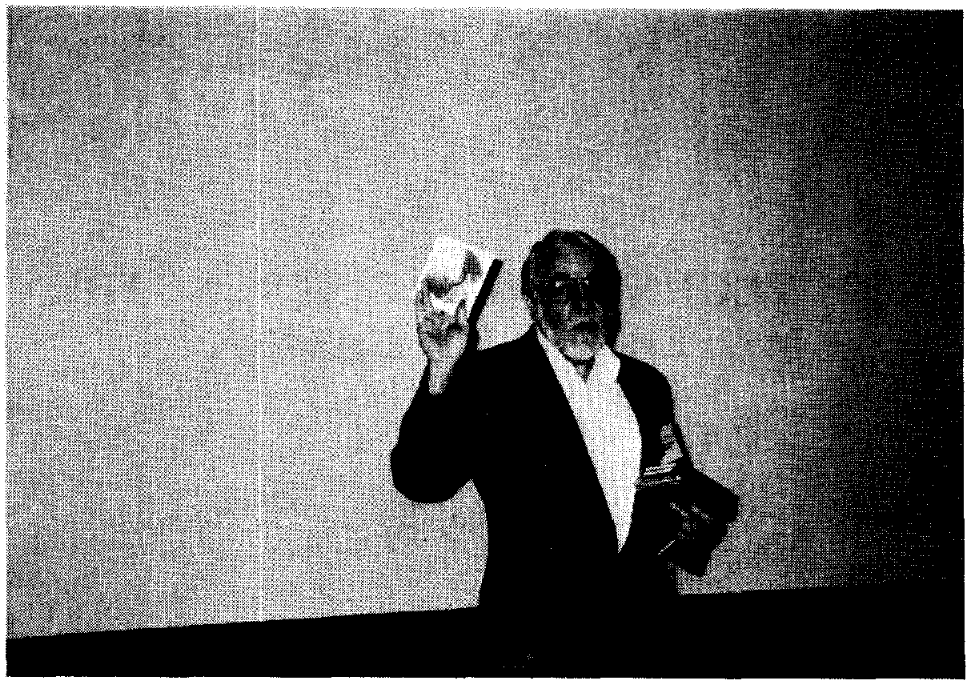


NIST

CDROM TECHNOLOGY TO STRENGTHEN MATERIALS EDUCATION

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ABSTRACT: It's a material world! Experiments and developments in the engineering materials science and technology field, presented at the National Educator's Workshops, are a valuable resource which can be used to enhance the teaching of this pervasive subject. In this presentation we will describe how selected experiments from the NEW proceedings can be published in digital format and distributed on CD-ROM. Faculty will be able to search for experiments by material name, by process, or author, for example. They will be able to download experiments, edit and print procedures along with tables and illustrations. Using CD ROM technology, the NEW experiments can become a first-rate, reusable laboratory resource for university faculty and teachers in high school and post secondary institutions.

PREREQUISITE: A desire to use the NEW Proceedings material in your teaching.

PURPOSE: To make the National Education Workshop Standard Experiments in Engineering Materials, Science and Technology more accessible to faculty and students using a digital format to supplement the hard copy book. This digital format will provide rapid access to experiments, including tables and graphics, through powerful text searching and hypertext linking. The intended media is CD ROM which allows an economical and thus wider distribution to the educational community.

THE NEW: UPDATE COMPENDIUM

WHO?	The Materials Education Council, in cooperation with the American Society for Engineering Education will review and select experiments to be presented in electronic format.
WHAT?	50 Experiments or more will be selected from seven years of NEW Proceedings.
WHERE?	The CD ROM will be produced in participation with Pennsylvania State University's Materials Research Laboratory which publishes the Materials Education Council's *Journal of Materials Education,* and other publications and teaching aids.

THE RESOURCE: EIGHT YEARS OF EXPERIMENTS AND DEMONSTRATIONS

The inventory is building.

Participation in NEW:Update Workshops

Attendance at Workshops							
1986	1987	1988	1989	1990	1991	1992	1993`
45	54	42	82	69	80	67	106*

* These 106 academic participants represented 50 separate institutions

Number of Experiments and Demonstrations Published in the NEW:Update Proceedings

Number of Experiments and Demonstrations								
1986	1987	1988	1989	1990	1991	1992	1993	Total
*	*	16	24	31	32	31	38	172

* Only updates on developments in the materials science field were presented in 1986 and 1987.

DIGITIZED TEXT DATA REPRESENTATION, STORAGE and RETRIEVAL

This subject is key to understanding why the soft copy of the NEW Compendium requires digitized text submission, such as that produced by a word processor. This form of data enables the most powerful search capability for data retrieval. Digitized text may also be produced from hard copy by using a scanner equipped with optical character recognition (OCR) software.

By contrast, a graphic page image can be used to store text and graphics data. In this case, a scanner is used to capture an entire document page as a grid of black and white pixels. OCR software is not used. Text, as well as drawings, pictures, and tables are represented as part of the same page image. Captured this way the text is not machine-readable and therefore not searchable. Only the whole page can be indexed and accessed. This approach may be useful where the arrangement of the text on the page is complex, where graphics appear with regularity, and when the user can find the desired page by means of a key word or hierarchical index that points to the page image. Graphic page storage also requires substantially more space than digitized text.

Text data may be structured by an outline for browsing purposes. Users can select chapters, sections, and headings as convenient access points, provided they know that the material of interest is located within that part of the text.

Key word searches provide another way of accessing text. Users can find information when its location is not obvious, or when its occurrence is so frequent that looking though a book index becomes tedious or impractical. Key terms may be combined to find unique "and" , "or", and "not" related documents.

Besides the above search methods, the software to be used in the Compendium CD-ROM will provide hypertext capability. Hypertext describes different ways of linking related items to each other. In its simplest form, know as "sideways browsing," the user selects a displayed word or phrase and with a single keystroke finds all documents that contain that word or phrase. It is possible to search for related terms as well, or provide for item linking created by the publisher or user. "Fuzzy logic" searching is also provided. This permits detection of "near hits" and ranking by probability of value of the hits.

Graphics, which includes pictures, charts, illustrations, and formulas, will be searched in the Compendium by means of indexed captions.

The user may make soft copy "notes" as review of the material proceeds, for later use.

In digitized form, including the essential graphics charts and pictures, the Compendium will take an estimated 1/6 of a CD-ROM's capacity. This will leave adequate space for expansion in future years and for additional still and motion video.

PROJECT ACTIVITIES REQUIRED to PRODUCE the COMPENDIUM CD-ROM

SET UP

A project management system will be set up for reporting activity and maintaining essential files.

USER NEEDS

"Users" are the ultimate beneficiaries of this project. Faculty who have been active in the workshops and in use of the proceedings will be consulted prior to drafting specifications for the end product, insofar as these requirements can be achieved in this initial issue. Some requests may be set aside for future consideration. By the end of this activity, agreement will be reached on the experiments to be included in the compendium; specifications for searching, reading, and using the information; and the platform(s) for this initial issue.

SOFTCOPY BOOK DESIGN

A style sheet will be prepared that reflects the user specifications for searching, reading, and using information. A production guideline will be prepared outlining the sequence and procedures for data preparation and digitizing. The guideline will accommodate the different types of documents, including tables, graphics, photographic images, etc. The guideline will include quality control processes to ensure accuracy of the final product.

TEXT and GRAPHICS ENTRY

The difficulty of producing the compendium will be eased by obtaining electronic files of documents from their presenters, where they exist. However, we anticipate that initially some of the text and graphics content will not be found in digital form. Many documents and images will need to be scanned using optical character recognition (OCR) and graphics software. Student interns will check for errors in text scanning and pay meticulous attention to formulae. Formulae may be treated as scanned text or as graphics, whichever may best suit user needs.

Final text must be in one of the following formats: WP5.0 or 5.1, WP for Win 5.1 or MS Word for Win 2.0.

A log will be kept of all activity, including "lessons learned" and recommendations made for document style and format submitted in future Proceedings.

Project Activities continued)

BUILD AND TEST

The components of each experiment - text, tables, graphics, and images will be formatted in accordance with the previously defined style sheet. The compendium will be indexed and compiled using the IBM BookManager software.

Once compiled, the compendium can be tested while it resides on the hard disk. A developer's test will ensure that the book operates as expected. Test copies will be produced and distributed on 3.5" disks for loading on testers' hard drives. Selected users will then search, read, display, print and copy the information as it is intended to be used by faculty and students. When revisions are complete, the master file will be transferred to a "one-off" single CD ROM for final testing.

PRODUCE AND DISTRIBUTE

A vendor will be selected for production - stamping the discs, imprinting with art work, inserting into the familiar "jewel" box and sealing for distribution. Distribution of the CD ROM will be on an "at cost" basis to NEW:Update attendees, interested faculty, teachers and media centers of educational institutions.

The IBM BookManager DOS/Windows/OS/2 Read module will be included on the Compendium CD-ROM.

EVALUATION

Formative and summative evaluation will ensure acceptance of the initial issue and provide recommendations for future editions of the Compendium.

SUBMISSION GUIDELINES

Based upon experience with this first CD-ROM, a guide will be prepared to supplement the existing Call for Papers standards where changes would facilitate both hard copy printing and soft copy production.

RELEASE DATE

Project funding for the CD ROM has been requested from the National Science Foundation. Summer '95 is the anticipated release date if funding is granted.

POTENTIAL IMPACT

THE EXPERIMENTS

Each experiment that is submitted for presentation at the Workshops is subjected to peer review prior to acceptance. During presentation at the Workshop, experiments are critiqued on site and also videotaped for review by the author or other participants. This acceptance process ensures it's value as a laboratory resource.

LABORATORY RESOURCE

In their present form, the experiments are proving their value to faculty who have adopted them for instruction. The Compendium on CD ROM will widen this audience and provide the materials education community with current, valuable aids for teaching and research in a format that can be readily searched, retrieved, and edited. It is expected that the experiments will find their way into the laboratories of educators at every level.

NETWORKED RESOURCE

Once installed in a CD ROM tower at a media center, the Compendium on CD ROM can be accessed through local and wide area networks.

COMPENDIUM PROGENY

Some experiments will have added value when presented with video, animation, and interactive content. Future issues of the Compendium may include multimedia presentations.

This experience will allow the publication of a guide for submitting future experiments and demonstrations to provide efficient and rapid dissemination.

A production guide for faculty interested in developing similar materials in digital form will be a product of the lessons learned in producing this first CD ROM.

A COMPARISON of ESTIMATED PRINTING and CD-ROM PRODUCTION COSTS

This is for a quantity of 400 NEW Update93 Proceedings. The cost excludes one time costs, data preparation, editing and authoring costs.

HARD COPY			
PRINTING & BINDING	$9,600	Unit Cost	$24
SHIPPING			$ 3
CD-ROM			
PRODUCTION & PACKAGING	$3,200	Unit Cost	$ 8
SHIPPING			$ 1.50

THE FEDERAL GOVERNMENT is a LEADER in EFFICIENT DATA STORAGE, DISTRIBUTION and RETRIEVAL THROUGH the USE of CD-ROM TECHNOLOGY

On November 18, 1993, OPNAV Instruction 5230 was issued. This instruction establishes CD-ROM technology as "the preferred media for storage and dissemination of data and information" for the Navy and Marine Corps.

All other major agencies are actively engaged in the production of CD-ROMS in order to increase efficiency of storage and distribution of data. Some examples:

The Government Printing Office publishes the massive USCode on one CD-ROM for $30.

The US Coast and Geodetic Survey (USCGS), a leader in the field, publishes geographic data on CD-ROMs for industry and the public. The USCGS has took the lead in 1986 by establishing a Special Interest Group on CD-ROM Applications & Technology (SIGCAT). This group has sponsored many studies on the technology and applications and participated in international standards setting. Meetings open to the public and government agencies are held every two months around the country, often in the Washington Metropolitan Area.

The Department of Agriculture is using CD-ROMs to preserve irreplaceable old archived documents which are disintegrating. In this case, graphic page storage is used to preserve the many tables, figures and illustrations contained in the documents.

END of PAPER

CD-ROM Technology to Strengthen Materials Education

Robert Berrettini, Administrator, MEC
Evelyn McKenney, Instructional Designer
Alfred McKenney, Engineer

NEW:Update 94

An Opportunity

- A Resource to be Mined:
  - *Six years of NEW experiments*
  - *172 papers through 1993 - 2,000 pages*
  - *41 more papers in 1994*

NEW:Update 94

A Plan

- Select and publish a Compendium
- Approximately 50 experiments
  - *Broadest materials and process coverage*
  - *Widest audience*

NEW:Update 94

Management

- MEC and ASEE to manage
- Proposal submitted to NSF for assistance

NEW:Update 94

The Media Delivers:

- A resource for Laboratory Experiments
- A Resource for Student Investigations

NEW:Update 94

A Solution: Electronic "Soft Copy"

- Searchable
- Retrievable
- Editable
- Printable
- Transmittable

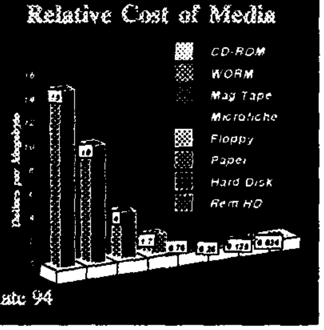
NEW:Update 94

Federal Agencies

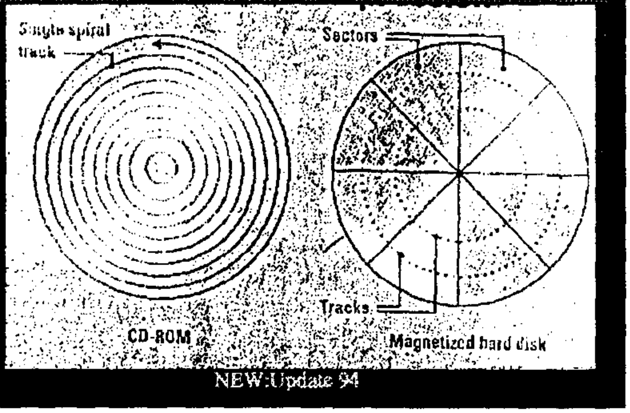
- US Naval directive
- US Dept of Agriculture
- US Coast & Geod Survey
- Library of Congress
- US Code
  - *30 volumes, 30,000 pages, $1200*
  - *1 CD-ROM, $34*

Vehicles for Delivery

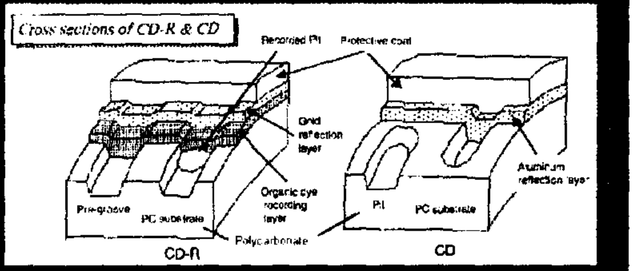
- Floppy, 1.5meg, ~600 pages of text
- Hard drive
- CD-ROM, 600mb, ~280,000 pages


Relative Cost of Media

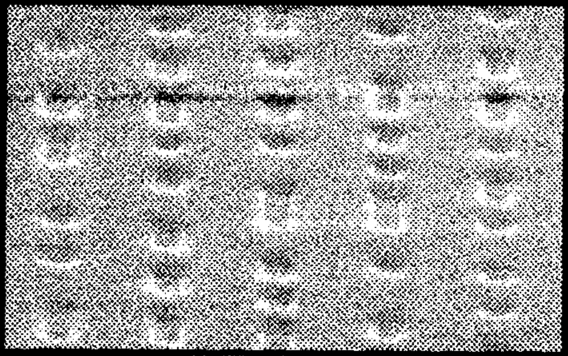
CD-ROM vs Mag



CD-ROM Structure


Cross sections of CD-R & CD

CD-ROM Surface



Demonstration of "Soft Copy"

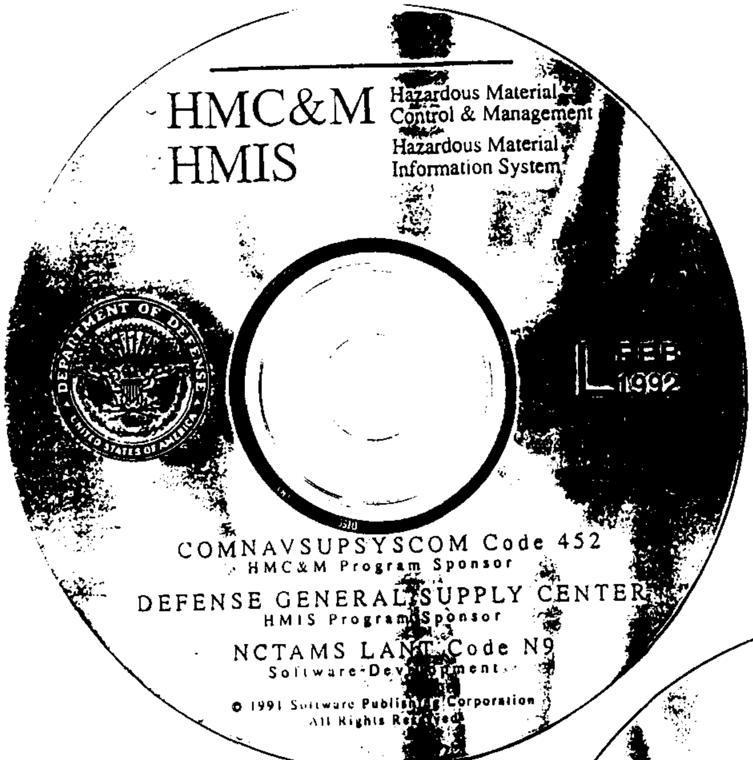
- 3 of the 1994 papers
- On hard drive
- Future:
  - *Still pictures: graphics or photos*
  - *Video & sound*
  - *Multi-platform*

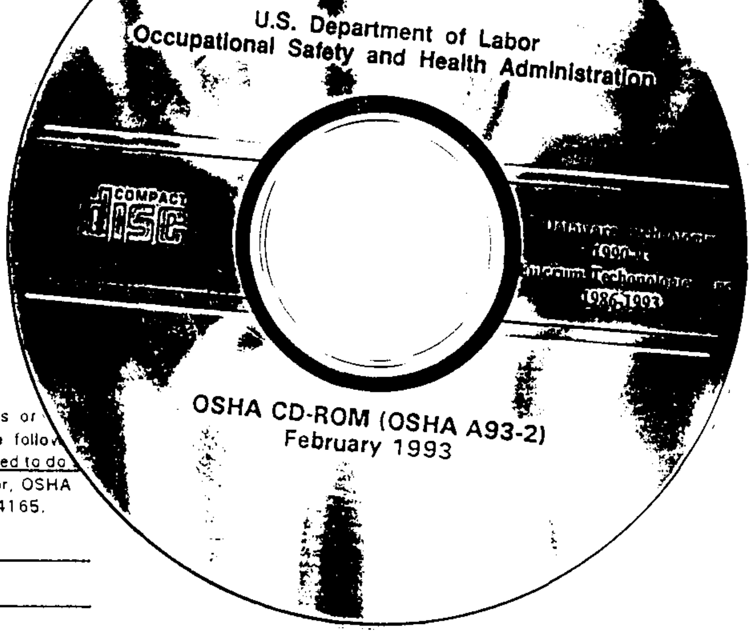
ABOUT the AUTHORS

Robert Berrettini has served for the past 14 years as administrator of the Materials Education Council and managing editor of the Journal of Materials Education. He has been directly involved in the development of video taped courses and teleconferences, and a variety of teaching aids in materials science ad engineering. He also currently serves as adjunct Assistant Professor in the Science, Technology and Society Program at Penn State. He received his B.A. (sociology) from Shippensburg University, M.S.Ed. from Wilkes University and D.Ed. from Penn State.

Alfred E. McKenney, a NEW Workshop participant since 1988, was a systems designer with IBM until he was assigned by IBM on its faculty loan program, to the School of Technology, Norfolk State University. At NSU he joined Dr. James Jacobs in a successful effort to fund and establish the School's Technology Education Learning Lab. This lab was a 20 station networked facility with CD-ROM, interactive videodisc, and video capture capabilities for multimedia development. Mr. McKenney instructed educators of all levels in the use of commercially available experiments programs such as the IBM Personal Science Laboratory, and in the production of their own materials. Mr. McKenney received his B.S. (engineering) from the United States Coast Guard Academy and his MBA from Harvard Business School.

Evelyn David McKenney, has ten years experience in interactive videodisc instructional design and production - for industrial training. She supervised a development lab responsible for converting 60 videodiscs from their original barcoded format to run on SONY, JVC, IBM, VISAGE, and ITS platforms. She has worked with UNIX, Apple, and DOS operating systems. As a senior consultant to Boeing Computer Services she designed and supervised their RCAS award winning prototype, an Open Systems Computer Based Training module for delivery at the desktop. The course was developed and delivered in an Open Systems, X-windows, SCO-UNIX networked environment. Ms. McKenney received her B.A. (design) from Antioch College and her M.Ed. from Harvard University.





OSHA A93-2
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I have the following comments/suggestions:

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EFFECT OF RISERS ON CAST ALUMINUM PLATES

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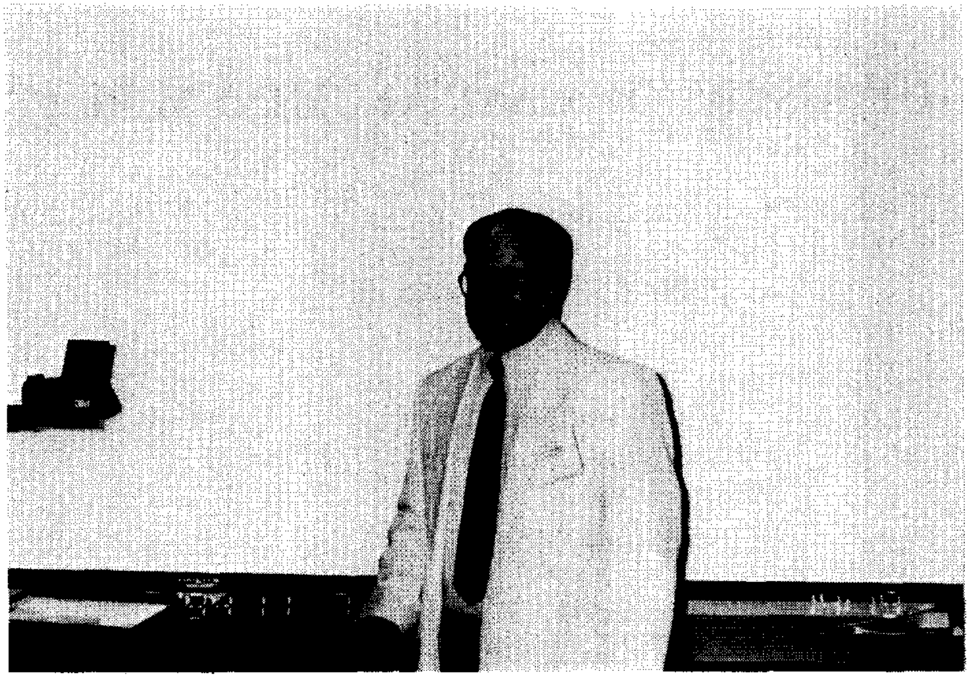

EFFECT OF RISERS ON CAST ALUMINUM PLATES

by
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KEY WORDS: Casting, risers, gating systems.

PREREQUISITE KNOWLEDGE: This experiment is intended for an introductory casting class. The students should have been exposed to the concepts of solidification shrinkage and risers in class.

OBJECTIVES: To demonstrate the solidification shrinkage of metal and common methods of producing a good cast part. To demonstrate the casting of a plate with no riser, a small riser, a medium riser, a large riser, and the effects of feeding distance.

EQUIPMENT AND SUPPLIES: To perform this experiment with aluminum or copper, a furnace capable of melting these metals is required. Crucibles, patterns, green sand or oil sand, flasks, and mold making tools are needed. A well ventilated area is recommended.

BACKGROUND Cast metal parts shrink in three stages: when cooling from the pouring temperature to the solidification temperature, during solidification, and from the solidification temperature to room temperature. The pattern maker makes the pattern oversize to compensate for the solid shrinkage but the gating system must compensate for the liquid shrinkage. In some cases, the runner system provides enough liquid mass but, in many cases, a riser is needed to compensate for the liquid shrinkage. A riser is a liquid reservoir closely connected to the casting and which is designed to be the last part of the system to freeze.

PROCEDURE: The kidney shaped pattern used for this experiment at Stout is shown in Figures 1 and 2 but any plate-like shape of the same approximate dimensions could be used. The pattern contains a draft (taper) on the sides so it could be removed from the mold after ramming. A small hole in the middle of the top of the pattern was used with a screw to help remove the pattern from the mold.

Eight mold configurations are used:

1. Gating into the middle of the plate with no riser,
2. Gating into the end of the plate with no riser,

3,4. Gating into the same locations using risers approximately 38 mm (1.5 inches) in diameter and 38 mm (1.5 inches) high.

5,6. The same gating locations using risers approximately 47.6 mm (1.87 inches) in diameter and 51 mm (2 in) high,

7,8. Gating from the same locations using risers approximately 63.5 mm (2.5 in) in diameter and 63.5 mm (2.5 in) high.

The riser patterns are shown in Figure 3. The risers should be placed as close to the plates as possible allowing for relatively easy removal. Riser bulbs were cut by hand under the patterns.

After the molds were made and vented, they were moved to a proper pouring area near the melting furnace. Weights were placed on top of the molds to prevent the cope from floating and a runout occurring. The aluminum was poured in two melts at 680°C (1250°F). Once the castings solidified and cooled, they were shaken out.

ANALYSIS OF THE RESULTS: The amount of shrinkage was observed as a function of the presence and location of the risers. There was no shrinkage with the medium or largest risers when the gates were properly made as shown in Figures 4. It can be seen that the medium riser contains a shrinkage cavity indicating proper feeding. The large riser had a small hole on the far side and the shrinkage occurred internally. Figure 5 contains an example of the casting with a small riser with severe shrinkage on the bottom. Figure 6 contains an example of the casting with no riser, again with bottom shrinkage. Figure 7 contains a casting from another class with no riser demonstrating top shrinkage.

The importance of a proper gating system is shown in Figures 8 and 9. Figure 8 contains an example of a casting with a medium riser where the gate was not completely open. Figure 9 contains a similar example for a casting with a large riser.

Figure 10 contains a summary of the types of shrinkage observed in the other cases. At gate location 1 with no risers, a large shrink cavity was generally observed as shown by location "a" and was often at the bottom of the part. The shrinkage is at the bottom of the part when the hottest metal enters the part along the bottom and the top freezes first. Shrinkage from a gate at location 2 with no risers was generally at locations "b". Again, these are often at the bottom of the part.

The medium riser size is a borderline situation. If the gate into the riser and the riser connection are large enough coupled with a somewhat hotter pour, there was no shrinkage. Otherwise, smaller amounts of shrinkage occurred at "c" or "b".

The smallest risers were not adequate to feed without some shrinkage occurring and the castings with no risers had increased shrinkage.

It should be noted that modifications of the gating system may cause the hotter metal to flow along the top and the resulting shrinkage to occur there. Changes to a more simple plate-like shape may also change the location of the shrinkage.

The final observation was the relative amount of good casting versus the total amount of metal poured. The gating systems were removed and weighed, the cast parts were weighed, and relative amounts calculated:

% yield = 100 x (wt. of part/total wt. of part and gating system)

The major decrease in yield as risers were added are shown in the table:

Configuration	Yield
No risers	49%
Smallest riser	41%
Medium riser	40%
Largest riser	31%

The best configuration is the one with the smallest gating system that produces consistently good parts which, in this case, is the medium riser configuration.

SAFETY CONSIDERATIONS: The use of high temperatures and the presence of molten metal make safety a major consideration. Melting should be done in a well ventilated area. All furnace charge material should be dry and clean; this is particularly important for all metal added to the molten metal or a steam explosion may occur.

All personnel directly involved in pouring should wear protective clothing including face shields. All others should stay well clear of the pouring area in case a mold blows back.

The castings should be allowed to cool completely after pouring to avoid burns. Overnight cooling will guarantee to castings are cool enough to handle. If the castings are shaken out before they are completely cool, they should be handled with tongs and quenched until cool.

INSTRUCTOR'S NOTES: The instructor should demonstrate mold making using the type of mold material available before beginning to melt the metal, possibly in a previous class period. The students can then make their molds while the metal is melting with each student or group of students using a different mold configuration. A normal class size at Stout is about 25 students using eight molding stations. This allows some duplication in mold configurations. There are usually two sections on consecutive days so two replicates of each configuration are made.

A second demonstration may occur at this time if there are enough molds compared to the amount of metal melted to require a second crucible of metal. The first pour can be made at the recommended pouring temperature (approximately 680°C (1250° F) for aluminum) and the second pour can be made considerably hotter (approximately 780°C (1400°F)). The lower temperature pour should result in a relatively smooth surface while the hotter poured surface should be much rougher due to the hotter, more fluid metal seeping between the sand grains.

The size of the required riser can be approximately calculated using a number of techniques. For the current configuration, calculations were made using the modulus method:

$M = V/S$ where M is the modulus, V is the volume, and s is the surface area.

The modulus of the riser should be at least 10% greater than that of the casting to ensure that the riser freezes last. The volume of the casting, in this case, was approximated as a rectangular plate 130 mm x 78 mm x 18 mm (5 in x 3 in x 0.75 in). For the sake of easier calculations, a domed cylinder with a height equal to the diameter was considered.

$V = 130(78)(18) = 182520 \text{ mm}^3$
$S = 2(130)(78) + 2(78)(18) + 2(18)(130) = 25896 \text{ mm}^2$
$M_c = V/S = 182520/25896 = 7.05$
Assume the riser modulus to be 10% greater
$M_r = 1.1(7.05) = 7.76$
Assume the riser is a domed cylinder
$V_r = \pi(D^2/4)H + (1/6)\pi D^3$
$S_r = \pi DH + \pi D^2$
Assume D = H
$M_r = ((\pi/4)D^3 + (\pi/6)D^3) / 2\pi D^2$
$M_r = 0.208D = 7.76$
$D = 37.3 \text{ mm} \approx 1.47 \text{ in}$

The calculations gave a riser diameter of approximately 37.3 mm (1.5 inches). An abbreviated method shown in reference 1 for a truncated cone results in the same value. This would indicate that the small riser should be sufficient. Closer agreement could possibly be obtained by calculating the true volume and area but would still be approximate since various alloys can be expected to react somewhat differently. These calculations are generally considered to be used as a guide and serve to reduce the amount of trial and error needed.

REFERENCES;

1. American Foundrymen's Society Cast Metals Institute, Basic Principles of Gating and Risering, 1973, AFS, Inc, Des Plaines, IL, 60016-8399.

The tables containing the riser equations are contained in an accompanying Gating Worksheets document, page 24.

2. LaRue, J.P., Basic Metalcasting, American Foundrymen's Society, Inc., Des Plaines, IL. 60016-2277, 1989.

This is a very good introductory text on the techniques of making castings including safety precautions.

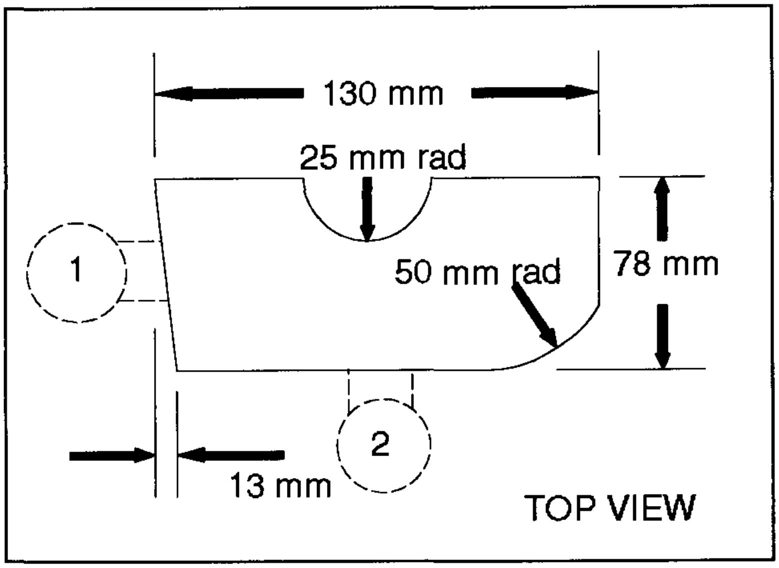

Figure 1: Top view of sample part showing riser locations.

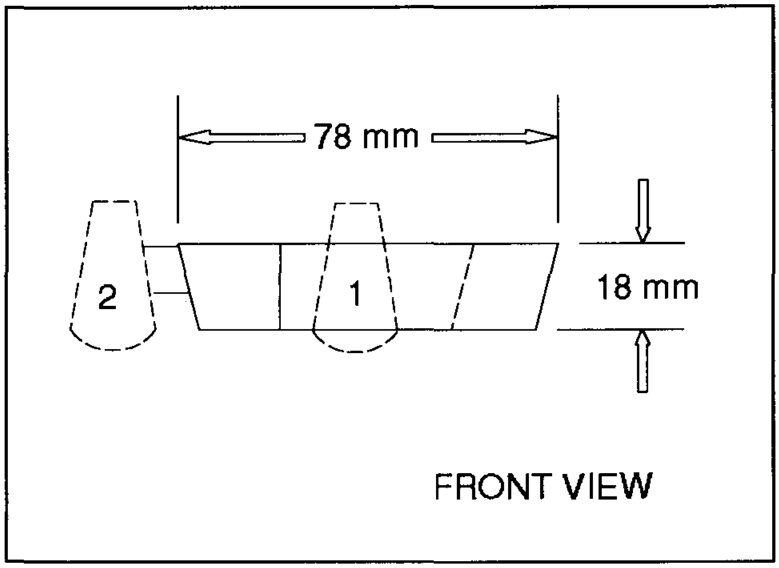

Figure 2:

Figure 2: Front view of sample part showing riser locations.

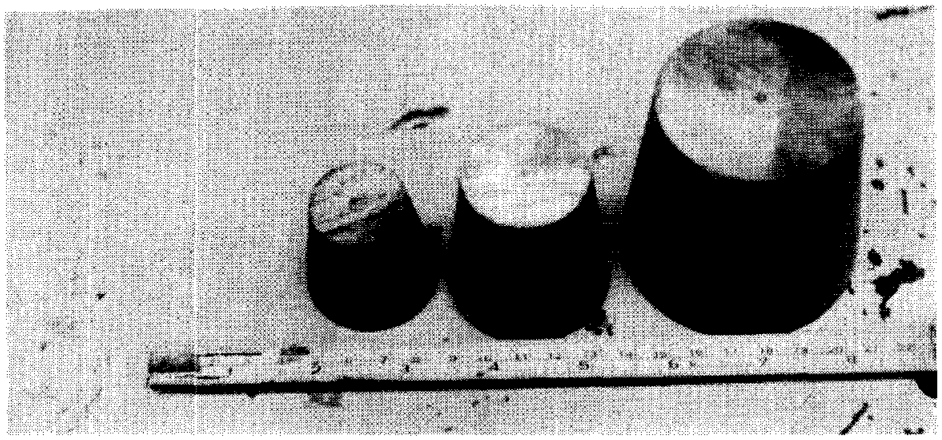

Figure 3. The patterns for the small, medium, and large risers are shown. The tape measure is in inches.

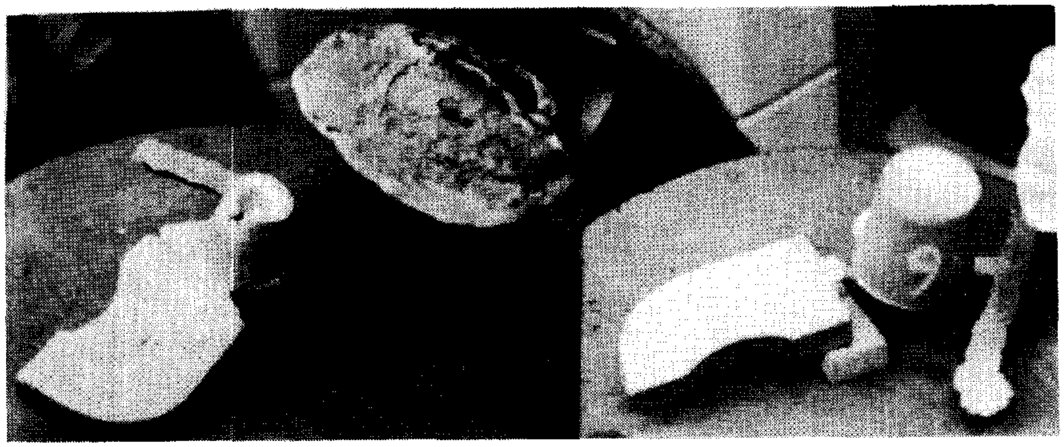

Figure 4. Successful end fed castings with a medium riser (showing the shrinkage cavity in the riser) and a large riser.

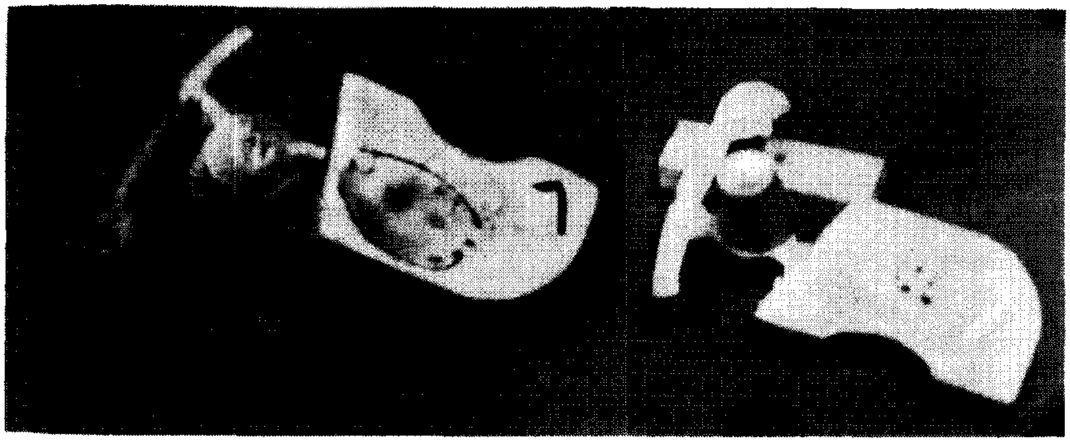

Figure 5. An end fed unsuccessful casting with a small riser and having bottom shrinkage.

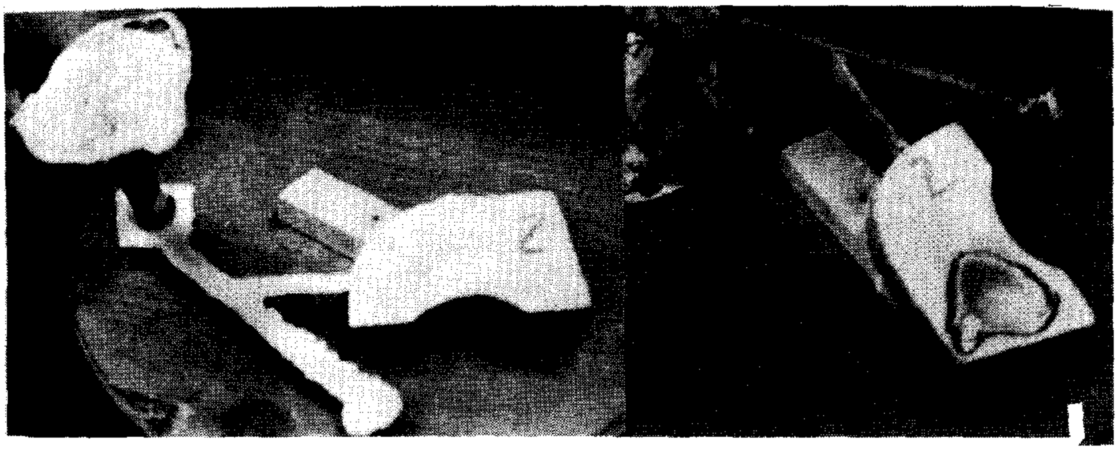

Figure 6. An unsuccessful casting with no riser and bottom shrinkage.

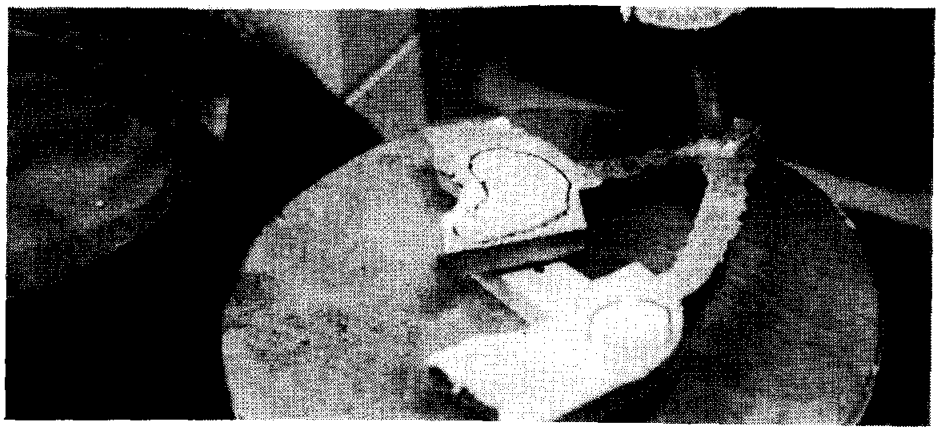

Figure 7. An end fed and a center fed casting with no risers and containing top shrinkage.

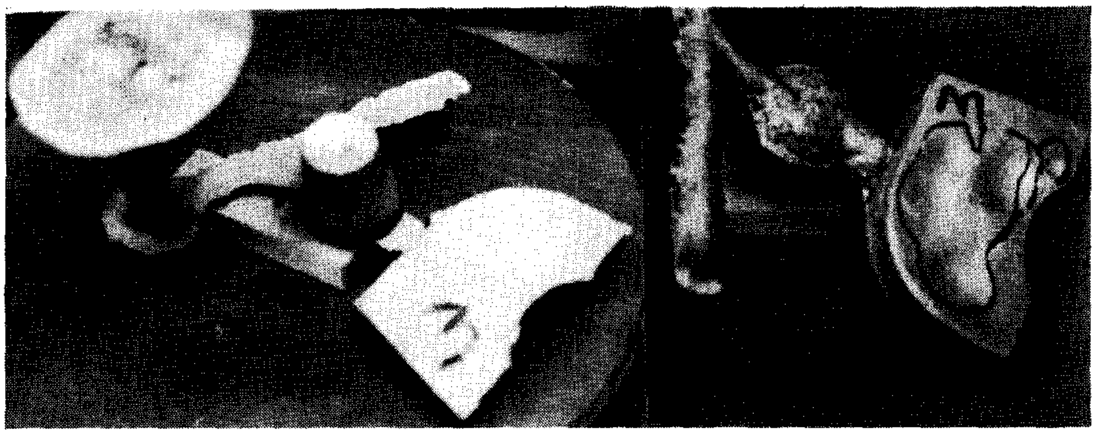

Figure 8. An unsuccessful casting using a medium riser and containing bottom shrinkage. The riser connection was not completely open and froze prematurely.

Figure 9. A incomplete casting with a large riser. This shrinkage was also due to the riser connection prematurely freezing.

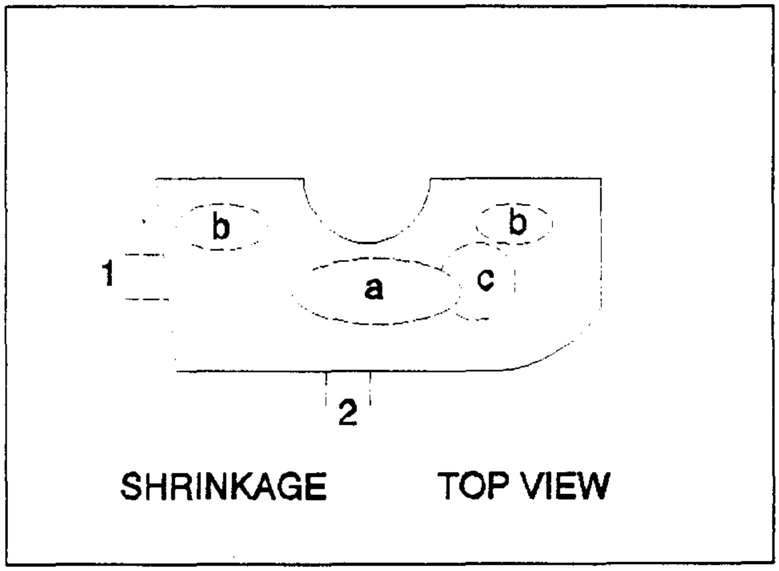

Figure 10: Schematic top view of part showing shrinkage locations.

EMERGING MATERIALS TECHNOLOGY

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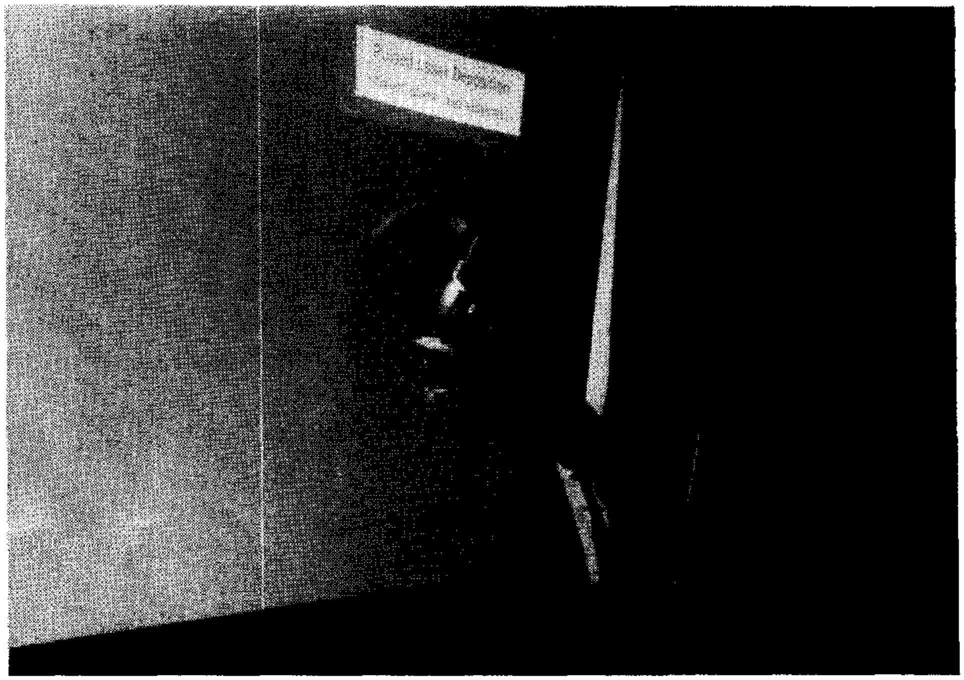

EMERGING MATERIALS TECHNOLOGY

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MATERIALS SHAPE THE FORM OF CIVILIZATIONS

MATERIALS ARE THE STUFF FROM WHICH THINGS ARE MADE

**MOST INDUSTRIAL NATIONS HAVE TARGETED MATERIALS TECHNOLOGY
AS ESSENTIAL TO ECONOMIC DEVELOPMENT AND COMPETITIVENESS**

"MATERIALS SHAPE THE FORMS OF CIVILIZATIONS".

"INNOVATION IN MATERIALS HAS BEEN RESPONSIBLE FOR MORE MAJOR TECHNOLOGICAL ADVANCES IN THE LAST 50 YEARS THAN IS GENERALLY RECOGNIZED. MATERIALS TECHNOLOGY IS TRULY AN ENABLING TECHNOLOGY. FURTHERMORE THERE IS NO SIGN THAT THE SOURCE OF INNOVATION IS DRYING UP".

"ADVANCED MATERIALS WILL BE THE RATE DETERMINING STEP IN THE INTRODUCTION OF TOMORROW'S NEW TECHNOLOGIES AND INDUSTRIES".

WORKING GROUP ON TECHNOLOGY, GROWTH
AND EMPLOYMENT-VERSAILLES ECONOMIC
SUMMIT, 1982

THE GROWTH AND ACTIVITY OF A WORLDWIDE MATERIALS SCIENCE COMMUNITY IS YIELDING A CORNUCOPIA OF POSSIBILITIES FOR NEW MATERIALS AND PROCESSES

THE PURSUIT OF THESE INTO TECHNOLOGY HAS LED TO SOME OUTSTANDING SUCCESSES - BUT ALSO SEVERAL EXAMPLES OF BITTER DISAPPOINTMENT OR EVEN BANKRUPTCY

LET US BOTH LOOK AT THE CORNUCOPIA AND EXPLORE HOW WE MAY MAXIMIZE THE POTENTIAL AND AVOID THE PITFALLS OF EMERGING MATERIALS TECHNOLOGY

THE CORNUCOPIA

STERIOSPECIFIC POLYMERIZATION

RAPID SOLIDIFICATION

MOLECULAR BEAM EPITAXY

PULSED LASER DEPOSITION

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ADVANCED COMPOSITES

- POLYMER MATRIX
- METAL MATRIX
- CERAMIC MATRIX
- CARBON CARBON

SUPERLATTICES

MOLECULAR COMPOSITES

DIAMOND COATINGS

NONLINEAR OPTICAL MATERIALS

CONDUCTING POLYMERS

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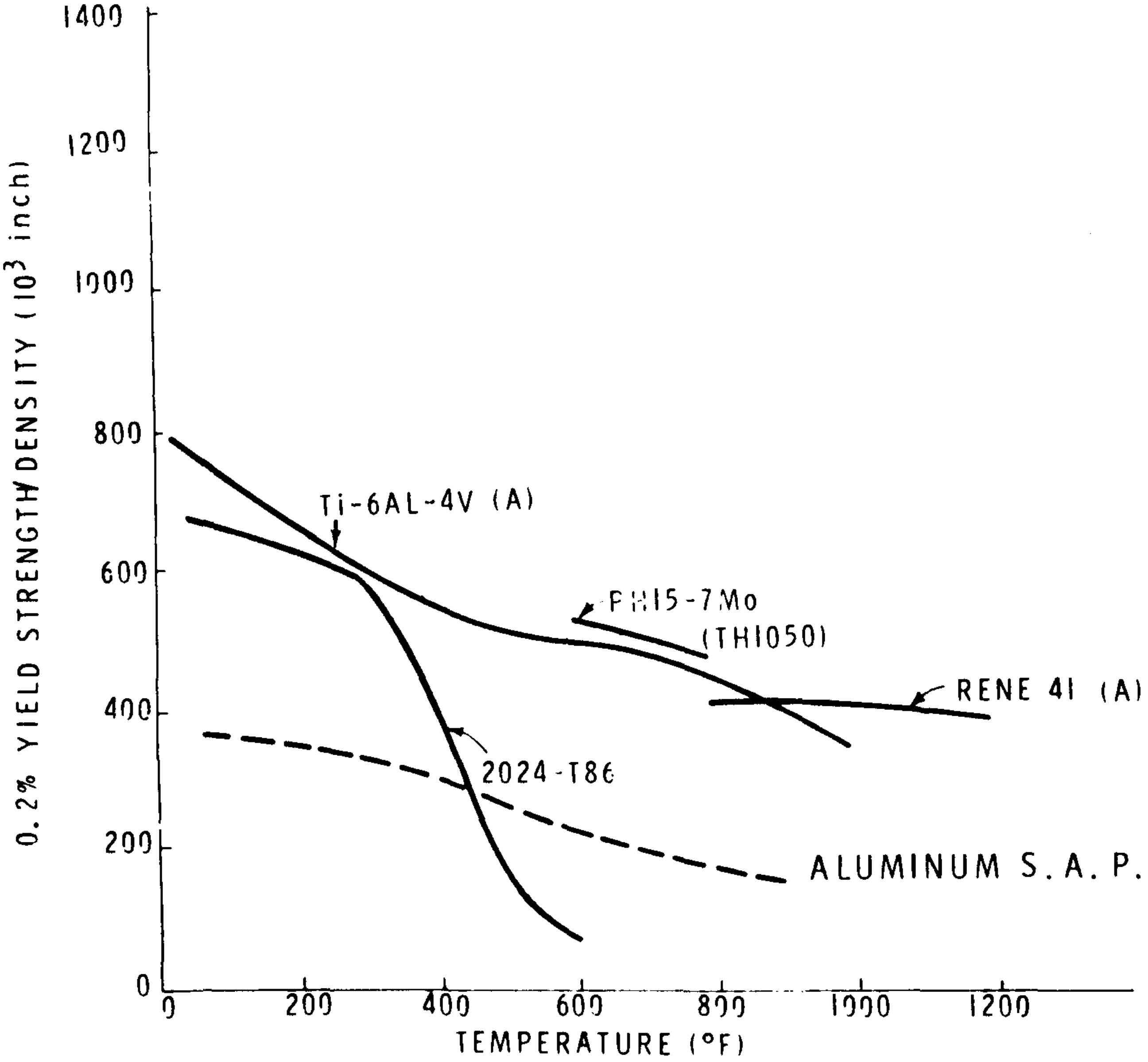
CONSIDER THE COMPETITION

THE DEVELOPMENT OF OXIDE-DISPERSION-STRENGTHENED ALLOYS IN THE 1950s AND 1960s WAS A METALLURGICAL BREAKTHROUGH. ALUMINUM AND NICKEL COULD BE ENDOWED WITH USABLE STRENGTH UP TO 90% OF THEIR MELTING POINTS INSTEAD OF THE FORMER TEXTBOOK LIMIT OF ABOUT 60%. YET THE DEVELOPMENT OF OXIDE-DISPERSION-STRENGTHENED ALLOYED PRODUCTS (E.G., SINTERED ALUMINUM POWDER) WAS NOT COMPETITIVE WITH NEW TITANIUM ALLOYS. SINTERED ALUMINUM POWDER AND RELATED PRODUCTS SOON WENT OUT OF PRODUCTION. DISPERSION-STRENGTHENED NICKEL WAS PERHAPS THE MOST SUCCESSFUL OF THE GROUP BUT NOT SUFFICIENTLY SO TO PREVENT SERIOUS FINANCIAL LOSS TO SOME OF ITS EARLY PROPONENTS. NOT ONLY DID COMPETITION EMERGE FROM OTHER APPROACHES TO HIGH-TEMPERATURE STRENGTH IN NICKEL-BASE ALLOYS, BUT IMPROVED DESIGNS FOR COOLING MADE THE ALLOYS' CAPACITY TO BE SHAPED INTO COMPLEX CONFIGURATIONS AS IMPORTANT AS STRENGTH. COMPETITION IS A FUNDAMENTAL RISK.



0.2% YIELD STRENGTH/DENSITY (10³ inch)
1400
1200
1000
800
600
400
200
0
0
200
400
600
800
1000
1200
TEMPERATURE (°F)
Ti-6AL-4V (A)
PH15-7Mo
(TH1050)
RENE 41 (A)
2024-T86
ALUMINUM S.A.P.

POLYMER MATRIX COMPOSITES

THE DEVELOPMENT OF POLYMER MATRIX COMPOSITES AND THE TRANSITION TO THEIR USE HAS BEEN ONE OF THE MORE EXCITING CHAPTERS OF MODERN MATERIALS TECHNOLOGY--BUT THERE HAVE BEEN PITFALLS. ROLLS ROYCE, IN COMMITTING ITSELF TO A JET ENGINE USING FAN BLADES MADE OF CARBON FIBER-EPOXY MATRIX COMPOSITE, DID NOT ADEQUATELY CONSIDER OR EVALUATE THE EROSION OR IMPACT-RESISTANT QUALITIES OF THE MATERIALS AND DESIGNS THEY WERE USING. BEFORE THE COMPANY COULD SOLVE THE PROBLEM, THIS OVERSIGHT LED TO ITS BANKRUPTCY.

WROUGHT TITANIUM

WROUGHT TITANIUM FOR USE IN JET ENGINES WAS A COMPARABLE DEVELOPMENT THAT PRECEDED THE ROLLS ROYCE EFFORT. SHORTLY AFTER ITS INTRODUCTION, A SERIES OF SERVICE FAILURES THREATENED ITS CONTINUED USE, BUT IN THIS CASE, A STRONG TECHNOLOGY BASE RESEARCH GROUP WAS EXPLORING SOME UNUSUAL BEHAVIOR IN TITANIUM, INCLUDING THE DETRIMENTAL EFFECTS OF HYDROGEN. THIS RESEARCH GROUP HAD THE BACKGROUND AND EXPERIMENTAL CAPABILITIES TO STUDY THE FAILURES AND QUICKLY REALIZED THAT SLOW STRAIN-RATE HYDROGEN EMBRITTLEMENT, WHICH WAS KNOWN TO OCCUR IN STEEL, COULD ALSO OCCUR IN CERTAIN TITANIUM ALLOYS. THE RESEARCH GROUP WAS ABLE TO PROVIDE SOLUTIONS TO ENSURE THE CONTINUED EFFECTIVE USE OF TITANIUM.

PROCESSING	⟷	PROPERTIES
Milling Time		Density
Pressed Density		Plastic Moduli
Firing Time		Strength
Firing Atmos.		Dielectric Const.
Firing Temp.		Permeability
.		Loss Factor
.		.
.		.
		.

PROCESSING	⟷	STRUCTURE	⟷	PROPERTIES
Milling Time		Composition		Density
Pressed Density		Phases		Plastic Moduli
Firing Time		Microstructure		Strength
Firing Atmos.				Dielectric Const.
Firing Temp.				Loss Factor

SYNTHESIS	⟷	STRUCTURE	⟷	PROPERTIES	⟷	USE
Milling Time		Composition		Density		Blade
Pressed Density		Phases		Plastic Moduli		Combustor
Firing Time		Microstructure		Strength		Heat Exchanger
Firing Atmos.				Dielectric Const.		Bearing
Firing Temp.				Loss Factor		Radome

Buessem - 1963

Burke - 1964

WHAT DO WE KNOW?

MICROSTRUCTURE - PROPERTY

HOW DO DIFFERENT CHEMISTRIES/MICROSTRUCTURES INFLUENCE PROPERTIES?

PROCESSING - PROPERTY

CAN WE SYNTHESIZE THE DESIRED MICROSTRUCTURE? UNIFORMLY AND REPRODUCIBLY?

CAN WE MODEL THE PROCESS TO GUIDE SCALE-UP?

PROPERTY-USE

WHAT PROPERTIES MEASURED IN THE LABORATORY WILL PREDICT BEHAVIOR IN SERVICE?

PROCESSING

A KEY ASPECT OF SUCCESSFUL TECHNOLOGY INVOLVES ROBUST PROCESSING - THE COST OF MAKING THE MATERIAL AND SHAPING IT INTO THINGS WE USE AND THE UNIFORMITY AND REPRODUCIBILITY OF THESE THINGS. THE EVOLUTION OF COMPUTATIONAL PLENTY IN THE LAST TWO DECADES NOW ENABLES US TO ATTAIN ADEQUATELY ELEGANT SOLUTIONS TO PREVIOUSLY INTRACTABLE SETS OF EQUATIONS WHICH DESCRIBE A PROCESS SUCH AS FORGING. THE MODELING AND COMPUTER SIMULATION OF THESE PROCESSES ALLOWS US TO DESIGN THEM QUICKLY AND RELIABLY, TO SCALE THEM AND TO CONTROL THEM. AN EXAMPLE TO BE GIVEN, OF NON-ISOTHERMAL FORGING OF A STEEL BILLET, WAS ACCOMPLISHED AS HIS SENIOR COLLEGE THESIS BY MY YOUNGEST SON. I SHOW IT NOT ONLY AS A PROUD FATHER, BUT MORE IMPORTANT TO MAKE THE POINT THAT AN INCREASING NUMBER OF YOUNG ENGINEERS ARE BEING GRADUATED WHO ARE FAMILIAR AND COMFORTABLE WITH THE SCIENCE AND TECHNOLOGY OF MODELING UNIT OPERATIONS AND UNIT PROCESSES. INDUSTRY WHICH LEARNS TO USE THEM WILL PROSPER IN A COMPETITIVE WORLD. THE CHEMICAL ENGINEERS HAVE DONE THIS FOR DECADES; IT IS RAPIDLY EXTENDING TO MANY OTHER FIELDS AND PROCESSES. EVEN WHERE THE PROCESSES ARE TOO COMPLEX FOR NUMERICAL SIMULATION - AS IN OUR CURRENT INABILITY TO FULLY MODEL THE MANY PROCESSES OCCURING IN AUTOCLAVE CURING OF COMPOSITES - A QUALITATIVE MODEL CAN BE DEVELOPED USING THEORETICAL OR EMPIRICAL KNOWLEDGE. THIS CAN BE EXPRESSED AS A SET OF RULES AS IN AN EXPERT SYSTEM OR OTHER ARTIFICIAL INTELLIGENCE BASED APPROACHES, WHICH BECOMES THE BASIS FOR MUCH IMPROVED CONTROL SYSTEMS.

DUAL PROPERTY Ti-6242 DISK

PROPERTIES ⟷ MICROSTRUCTURE ⟷ POTENTIAL WINDOWS

BORE

- TENSILE STRENGTH 180 KSI
- FRACTURE TOUGHNESS 70 KSI IN.
- LOW CYCLE FATIGUE 10^5N/100KSI

1750F/1750F/AC
ALPHA + BETA PROCESSED MICROSTRUCTURE

- SMALL ENGINE (HIGH RPM) DISK
- HIGH PERFORMANCE LARGE GAS TURBINE ENGINE DISK

RIM

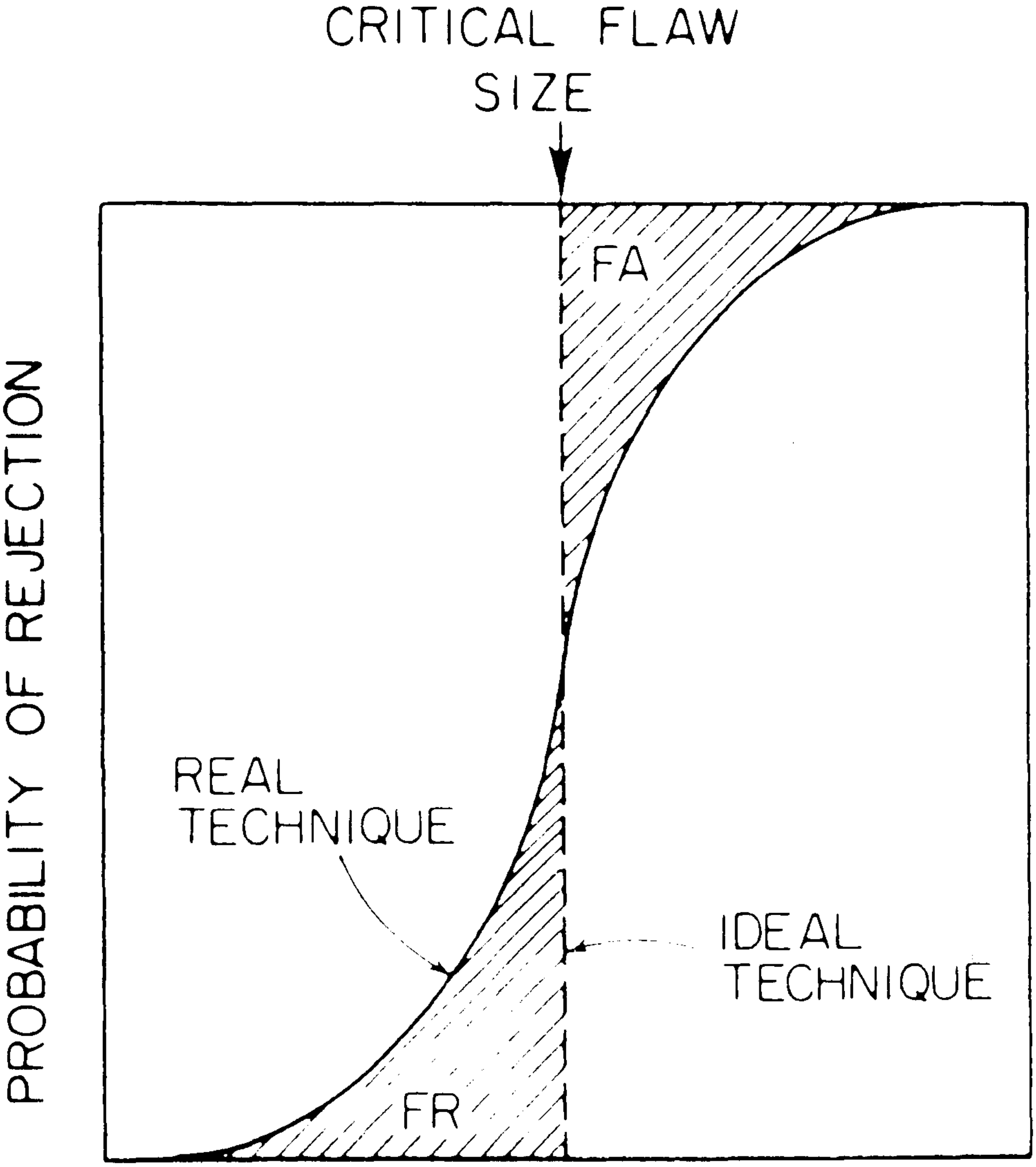
- CREEP PROPERTIES 95 KSI/.2%/120 HRS.
- FRACTURE TOUGHNESS 70 KSI IN

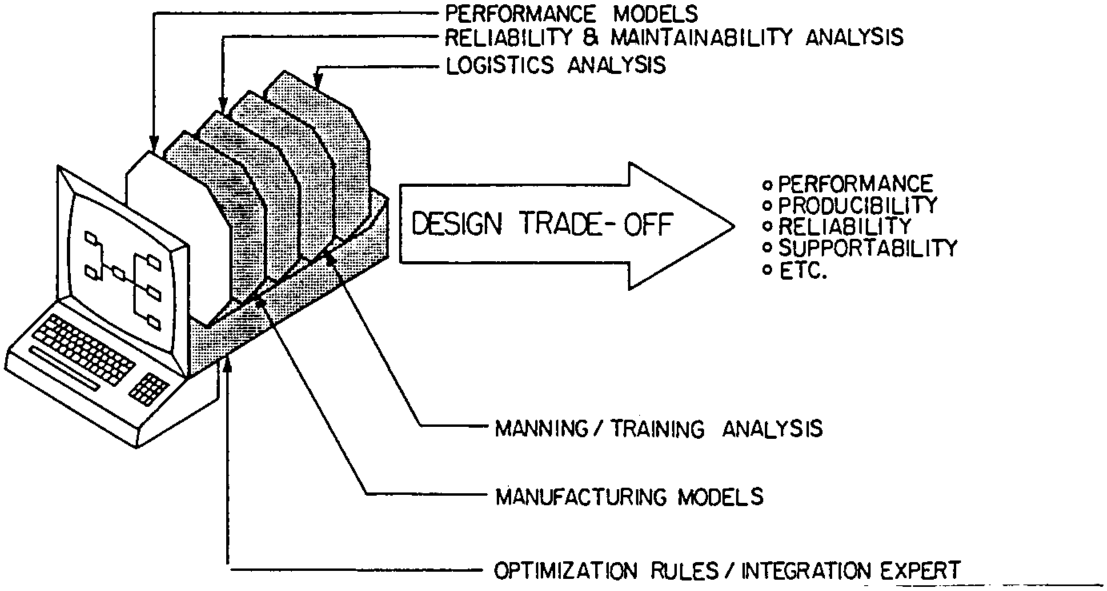
2100F/1840F/AC
WIDMANSTATTEN MICROSTRUCTURE

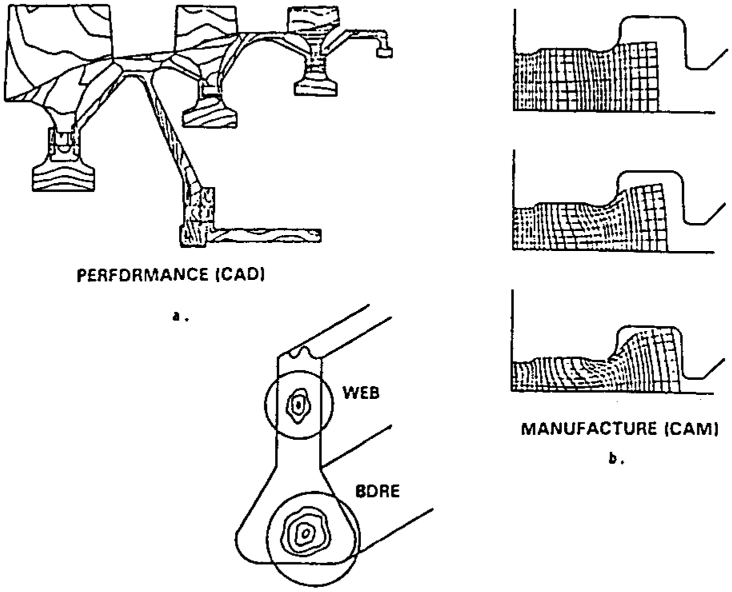
ROBUST CHARACTERIZATION AND DESIGN

INCREASINGLY, IN OUR TECHNOLOGICAL CIVILIZATION, WE ARE USING MATERIALS CLOSER TO THEIR LIMITS (TO ACHIEVE PERFORMANCE), AND THE CONSEQUENCES OF FAILURE ARE MORE CATASTROPHIC. THIS MAY NOT BE A NEW PROBLEM, BUT IT CERTAINLY TAKES ON ADDED IMPORTANCE WHEN THE VIABILITY OF AN ENTIRE INDUSTRY OR APPROACH TO SOLVING HUMANITY'S NEEDS - SUCH AS NUCLEAR POWER - DEPENDS ON HOW WE COPE WITH IT. NEW SCIENCE AND TECHNOLOGY, SUCH AS LIFE PREDICTION AND NONDESTRUCTIVE INSPECTION CAN ALLOW US TO ACHIEVE PERFORMANCE, EXTEND LIFE/REDUCE COST AND RETAIN RELIABILITY.

PROBABILITY OF REJECTION







PERFORMANCE (CAD)
a.

MANUFACTURE (CAM)
b.

SUPPORTABILITY (CAS)
c.

ASM EDUCATIONAL TOOLS NOW AND INTO THE FUTURE

Robert Uhl

Director of New Services Development
ASM International
St. Rt. 81
Materials Park, Ohio 44073

Telephone 216-338-5151

INFORMATION DELIVERY MODES
APPROPRIATE TO THE LIFE SPAN OF TECHNOLOGY

Emerging ↑	Personal Contacts
	Electronic Networks
	Seminars/Conferences
	Journals
	Magazines
	Conference Proceedings
	Online Databases
	Technical Books
	How-to Manuals
	Handbooks/General Reference Books
↓ Stable	MEI/Video

TLG/jld/modes

Longer-Term View: Holder of information will have power. What will the long-term hold for us "as users" can connect directly into databases? How do we position ourselves for the long-term ...

- Quality
- Quantity
- Structured
- Expert Systems (searchable)
- Consolidated
- More Searchable
- Ranking of Attributes

RCU4/jld/mkt2

"AN ELECTRONIC FUTURE"

Concept/Issues Planning Document

INFORMATION USERS

- Individuals
  - Research
  - Students
  - Engineers
  - Technicians
- University Libraries
- Corporate Libraries
- Research Laboratories
- Non-North American
- Non-English Reading
- CAD/CAM Producers
- Training Schools
- Universities
- Trade Associations/Societies
- Corporate Trainers

"AN ELECTRONIC FUTURE"

Concept/Issues Planning Document

PRODUCTS/SERVICES

- Traditional Book
- Segmented Print Product (print-on-demand)
    - Committee/staff-selected
    - Customer online selection
    - Online review of material/download
- Online/Electronic Product Search Engine (Tool)
    - Word, phrase, association, thesaurus, numerical values (properties), greater than/less than, etc.
- Expert System (online interface PC product, work station, LAN)
- Information Availability on Common Software (Lotus, Excel, etc.)
- CD-ROM
    - PC/LAN
    - download & print feature
    - single/multi-user
- Images
- Video
- Audio Tapes
- Bulletin Boards
- Segments of Database as Electronic Database Products
    - Selection as Segmented Print Product
    - Usage Issues such as for CD-ROM
- Information Central (complex vision -- multi-usage)

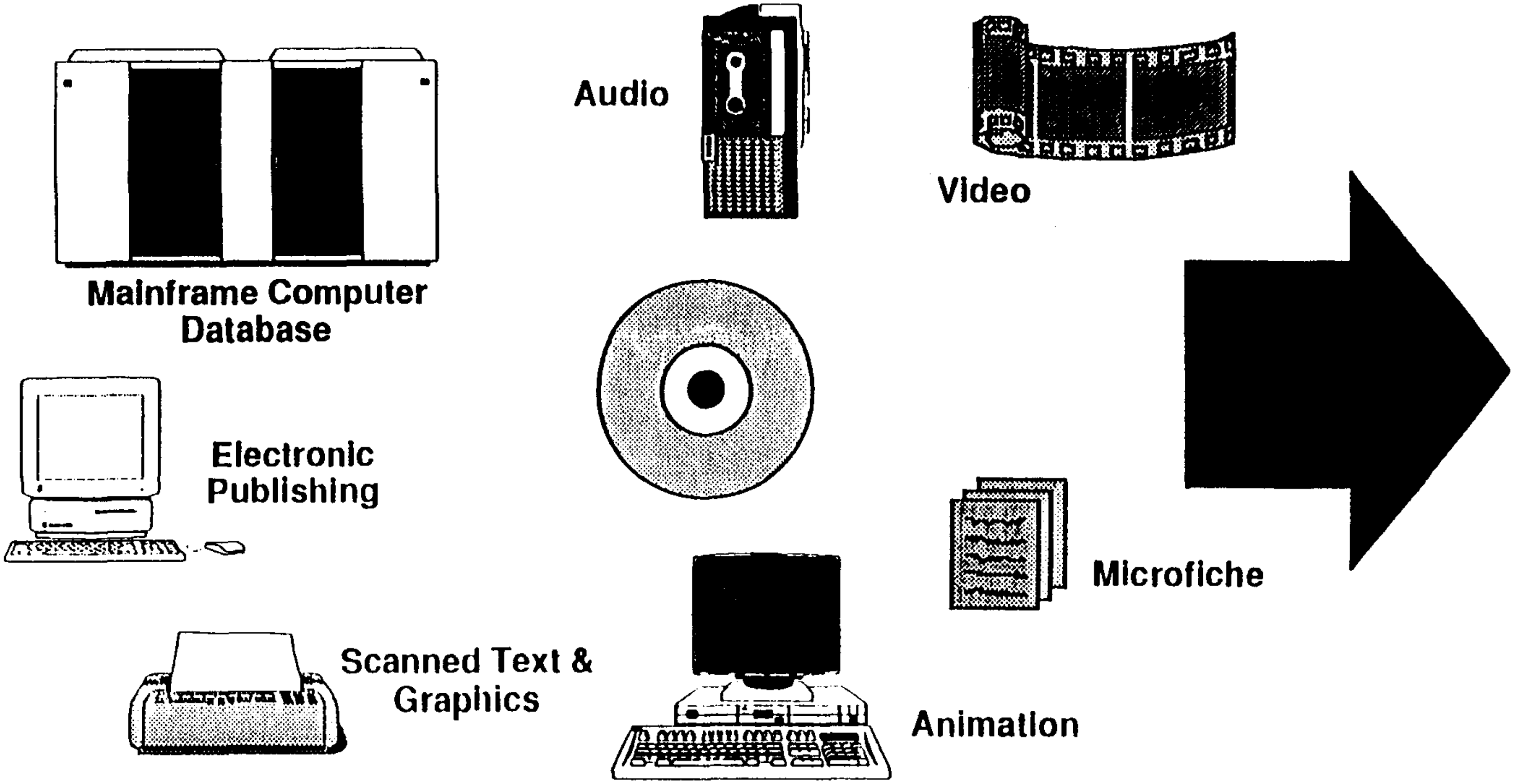
"AN ELECTRONIC FUTURE"

Concept/Issues Planning Document

DATABASE ARCHITECTURAL/BUSINESS ISSUES

- Online Purchase/Billing for Print-On-Demand Products
- Royalties on Online Downloads/Online Billing
- Online/Electronic Product Search Engines (Tools)
- Expert System (online interface, PC product, work station, LAN)
- Source of Material Information and Tracking (References -- original & last)
- Tagging System
- File Control
    - In-house/out-house
    - Security
    - Establishing updating programs on subscription products in electronic format
    - Maintenance
    - Copyright notices
- PC Download Online
- CD-ROM Royalties
- PC-Based Product
    - Networks
    - Site licenses
- Ability to Work Across Platforms
- Multimedia
- Database Contents
    - Text
    - Properties
    - Graphs
    - Artwork
    - Images
    - Audio
    - Video
- Bulletin Boards

Digital Info of Any Type on a Single Disc



Mainframe Computer Database

Audio

Video

Electronic Publishing

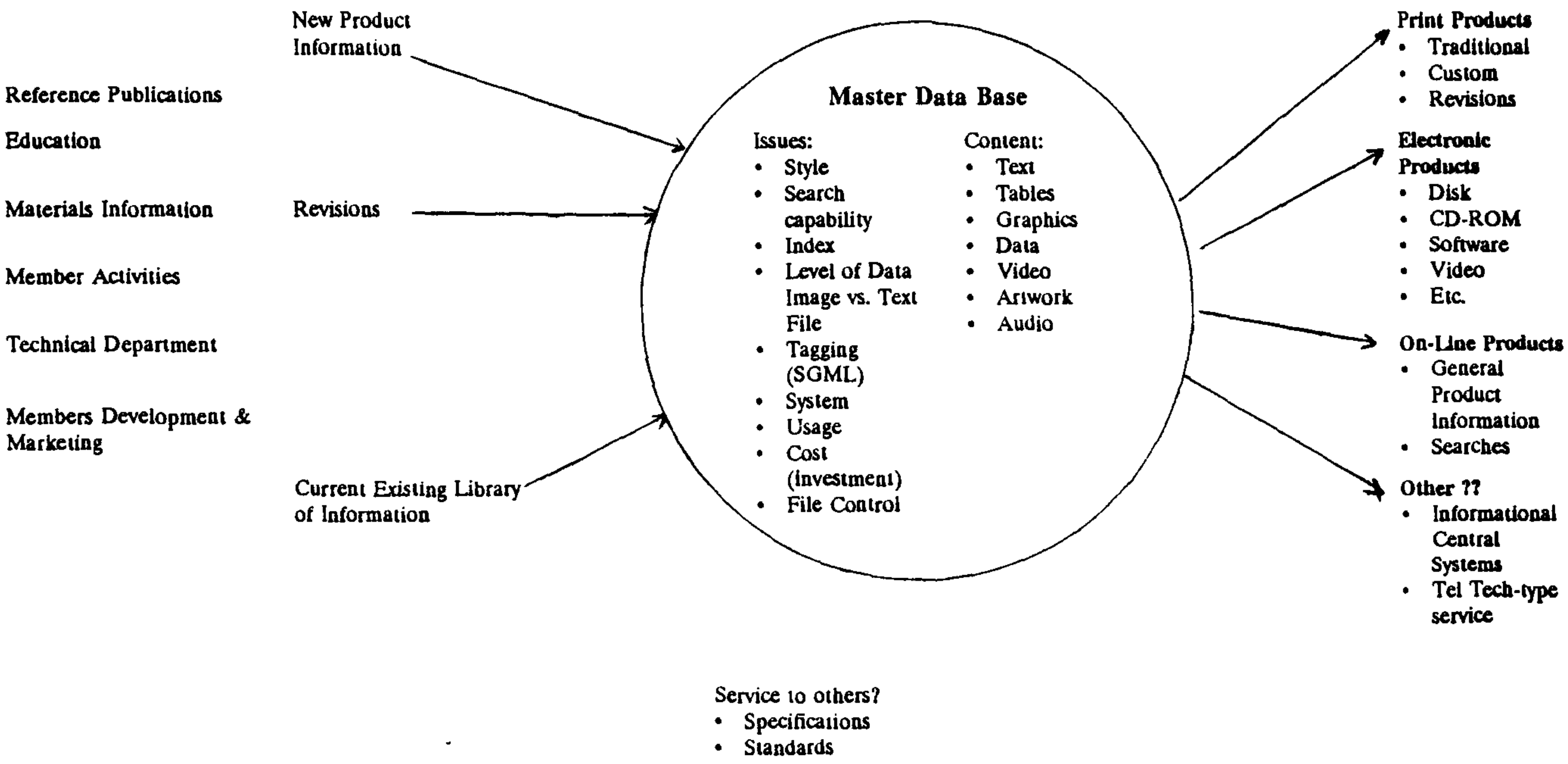
Microfiche

Scanned Text & Graphics

Animation

Master Database and Customized Products

An Organizational Perspective



Reference Publications

Education

Materials Information

Member Activities

Technical Department

Members Development & Marketing

New Product Information

Revisions

Current Existing Library of Information

Master Data Base

Issues:
- Style
- Search capability
- Index
- Level of Data Image vs. Text File
- Tagging (SGML)
- System
- Usage
- Cost (investment)
- File Control

Content:
- Text
- Tables
- Graphics
- Data
- Video
- Artwork
- Audio

Print Products
- Traditional
- Custom
- Revisions

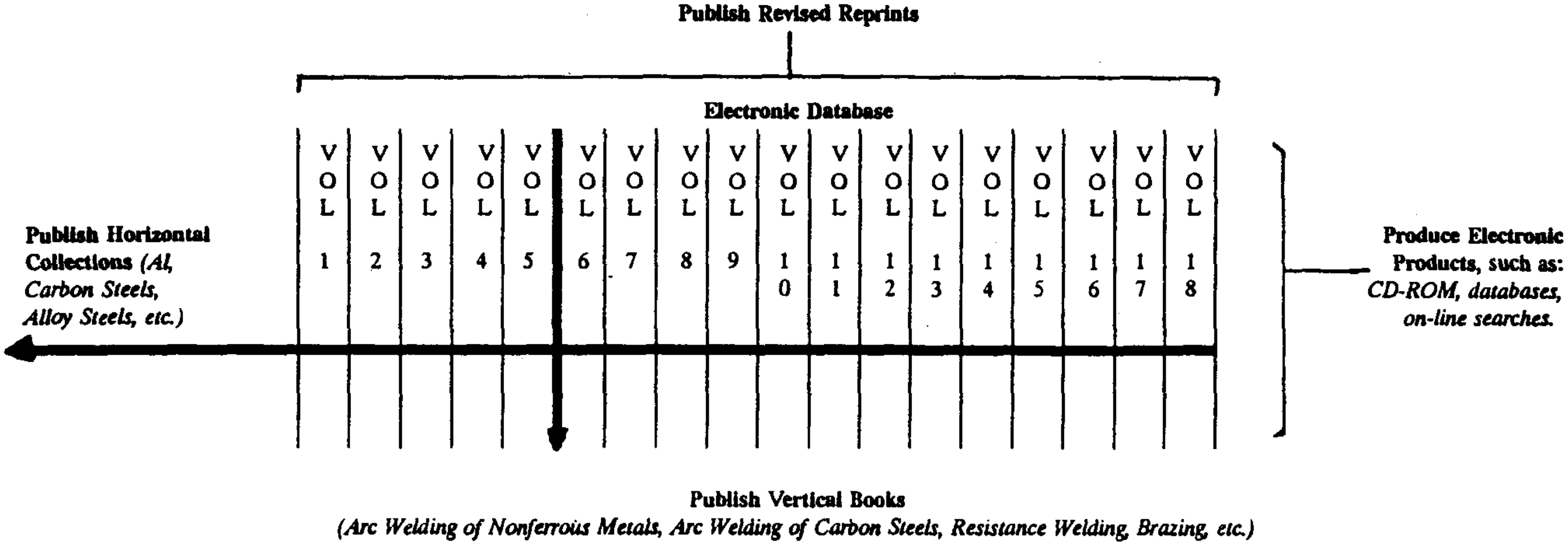
Electronic Products
- Disk
- CD-ROM
- Software
- Video
- Etc.

On-Line Products
- General Product Information
- Searches

Other ??
- Informational Central Systems
- Tel Tech-type service

Service to others?
- Specifications
- Standards

CONCEPTUAL OPERATION OF AN ELECTRONIC DATABASE RELATED TO PRODUCT DEVELOPMENT AND REVISION



<u>Review and Revision/Update of Electronic Database</u>
(Divisions, Committees, Authors)

Example: *Joining Division*

Review & Update Sections
1 Arc Welding of Carbon Steels
2 Brazing of Nonferrous Alloys
S —
S+1 —

KEY: *Revised sections would be reinserted into electronic database to be incorporated into updated reprints of Handbooks and horizontal/vertical products.*

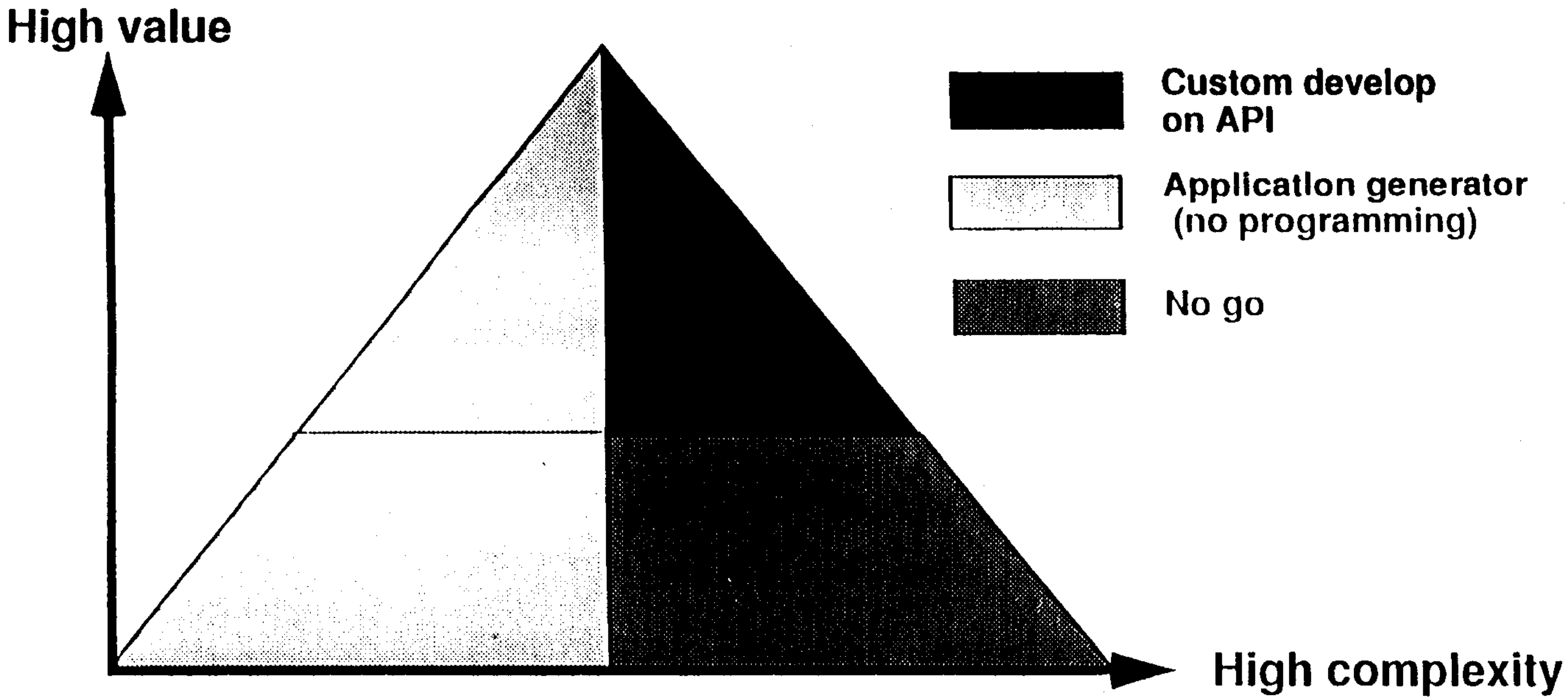
<u>Examples</u>:

- *Committee Revision* -- Publish revised update as a low-priced update series book and reinsert it into the electronic database.
- *Individual Author Update* -- Publish an expanded technical book, then edit it into a handbook sized article for its update and reinsert it into the electronic database. (Use divisional or committee review.)

RCU3/jld/edb

Title Development Options

Applications Value/Complexity Pyramid



High value

High complexity

- ■ Custom develop on API
- □ Application generator (no programming)
- ▨ No go

The Question??

How may these emerging technologies and new services benefit the educational community?

Image Based CD-ROM Working Library

- On-demand and available 24 hours a day on your campus network.
- Instructors review, select and print segments for classroom use and reference.
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DENSITY BY TITRATION

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Density By Titration

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Key Words: Polymers, density, polypropylene, titration, volumetric analysis, miscible, buret, displacement, percentage of error.

Prerequisite Knowledge: The student should have an understanding of the concept of density and be familiar with density calculations that involve data experimentally collected. Students should be given instructions on how to properly prepare and read a buret.

Objectives: 1) To better understand the concept of density. 2) To acquire volumetric analysis laboratory techniques and apply them to the determination of the density of polypropylene by titration and displacement.

Equipment and Supplies:

1. two 50 mL burets
2. buret clamp and stand
3. distilled water
4. ethanol
5. two 100mL beakers
6. balance
7. Bic round stic ® pen

Introduction:

Density is one of the deciding factors of a material's usefulness and selection for application. The concept of distinguishing between low-density and high-density polyethylene presented by Kolb[2] led to the development of this density lab involving volumetric analysis. Often called the flotation method, this procedure may be used to determine the density of a solid that is insoluble in two miscible liquids of different densities.[3]

The plastic barrel of a Bic[4] round stic® pen serves as the solid. It is composed of polypropylene and contains the pigment titanium dioxide, TiO_2. The density of the plastic, as reported by the Bic® Corporation, is .903 g/mL and must fall between the densities of the two liquids. Ethanol and water thus serve as suitable liquids.

Using titration, the end point is reached when a solution of the two liquids has a density equivalent to the plastic of the barrel. At this point, the plastic just becomes buoyant. The solution's mass and density can be calculated since the solution volume and volumes of both liquids at the end point of titration are known. The following procedure, previously described in the *Journal of Chemical Education* (1993, Vol. 70, p. 397), is used to determine density of the two liquids. Density of the plastic barrel is then determined utilizing titration and ethanol displacement. Sample data and calculated results are provided in the table.

Procedure A - Density of Ethanol and Water

1. Fill two 50 mL burets, one with distilled water and one with ethanol.
2. Label two 100 mL beakers, #1 and #2.
3. Mass beaker #1 and run 15.00 mL of ethanol into it from the buret.
4. Mass the beaker and ethanol. Save this for Procedure B.
5. Repeat the above procedure using water and beaker #2. Save this for Procedure B.

Procedure B - Density of Plastic by Titration

1. Remove and discard from the barrel the inside ink cartridge and the end caps of a Bic round stic® pen.
2. Use a sharp blade with **caution** to cut off two 3-4 mm pieces of the barrel. Save the remainder of the barrel for Procedure C.
3. Drop one piece into each of the two beakers containing liquids from Procedure A.
4. Observe evidence that the plastic's density is greater than ethanol but less than water (See the figure).
5. Place beaker #1, containing ethanol and plastic, under the buret with water. While stirring vigorously, titrate with water until the plastic just becomes buoyant (See the figure).
6. Record the volume of water required to reach the end point.
7. Remove the plastic carefully from the solution and use a graduated cylinder to determine the solution's volume.

Procedure C - Density of Plastic by Displacement

1. Determine the mass of the remaining Bic round stic® pen barrel saved from Procedure B.
2. Record the volume of ethanol remaining in the buret.
3. Drop the barrel into the ethanol buret and record the new volume.

Calculations

1. Calculate the density of the two liquids from the Procedure A data.

2. Calculate the mass of both liquids (M=dV) in the titration using the densities obtained in Procedure A. Add the masses of water and ethanol to determine the solution's mass at the end point.
3. Calculate the density of the plastic by using the solution's mass and volume. At the end point, the solution's density is equivalent to that of the plastic.
4. Calculate the percentage of volume reduction of the titrated solution.
5. Calculate the plastic density again, this time using the barrel mass and volume displacement of ethanol obtained in Procedure C.
6. Calculate percentage of error for the densities of water and ethanol. Calculate the percentage of error for the density of plastic determined by both titration and ethanol displacement.

Notes to the Instructor:

1. No open flames in the lab area should be permitted. Wear protective eyewear and aprons.

2. This exercise has been useful in advanced high school and introductory college chemistry classes. As a new twist to an old concept, it provides a vehicle for the early introduction of volumetric methods. Students apply the density data for the two liquids to determine the mass of the titrated solution and become aware that the total volume of the titrated solution is not the sum of the volumes dispensed from the two burets. After calculating the approximate 4% volume reduction, students should attempt to explain why the solution's volume is less than the sum of the volumes of the two liquids.

3. Percentage of error analysis is made on the densities of water, ethanol and the plastic pen barrel determined both by titration and ethanol displacement. Percent error for the plastic ranges from 0.00% to 6.0% by both methods, with ethanol displacement usually providing better results. Students are encouraged to suggest weaknesses of both methods that might account for errors.

4. The Bic round stic® pen was chosen because it is readily available, fits in the 50 mL buret, and is a common object in the lives of many students. Plastic containers imprinted with recycling code 4, low-density polyethylene (LDPE), and 5, polypropylene (PP), provide other sources of plastics whose density may be determined by titration using water and ethanol. Additional related studies can explore the separation and identification of various plastics with solvents of different densities as described by Kolb[5].

References:

1. Author to whom correspondence should be addressed.
2. Kolb, K.E.; Kolb, D.K.: "A Quick, Simple Demonstration to Distinguish between HD and LD Polyethylene." *J. Chem. Educ.*, Vol. 63, No. 5, May 1986, p. 417.
3. Craig, R.E.R.: "Determination of the Density of Crystalline Solids in the Undergraduate Laboratory." *J. Chem Educ.*, Vol. 66, No. 7, July 1989, pp. 599-600.

4. Bic is a trademark of the Bic Corporation, Milford, CT 06460.
5. Kolb, K.E.; Kolb, D.K.: "Method for Separating or Identifying Plastics," *J. Chem. Ed.*, Vol. 68, No. 4, April 1991, p. 348.

Source of Supplies:
Flinn Scientific Inc., P.O. Box 219, 131 Flinn Street, Batavia, IL 60510

Table: Sample of Experimental Data and Calculated Results

Procedure A	***Trial #1***	***Trial #2***
1. Volume of ethanol	15.00 mL	15.00 mL
2. Mass of ethanol	11.80 g	11.81 g
3. Calculated density of ethanol [2 ÷ 1]	.7867 g/mL	.7873 g/mL
4. Volume of water	15.00 mL	15.00 mL
5. Mass of water	14.93 g	14.88 g
6. Calculated density of water [5 ÷ 4]	.9953 g/mL	.9920 g/mL
Procedure B		
7. Volume of water used in titration	12.20 mL	12.50 mL
8. Volume of solution at end point	26.0 mL	26.0 mL
9. Calculated mass of solution [(7 x 6) + 2]	23.94 g	24.21 g
10. Calculated solution density and plastic density [9 ÷ 8]	.921 g/mL	.931 g/mL
11. Calculated volume reduction [(1 + 7) - 8] ÷ [(1 + 7)] x 100	4.4%	5.5%
Procedure C		
12. Mass of pen barrel	1.77 g	1.82 g
13. Volume of pen barrel	1.97 mL	2.00 mL
14. Calculated density of barrel by displacement [12 ÷ 13]	.898 g/mL	.910 g/mL
15. Percent error by titration	1.99%	3.10%
16. Percent error by displacement	.55%	.78%

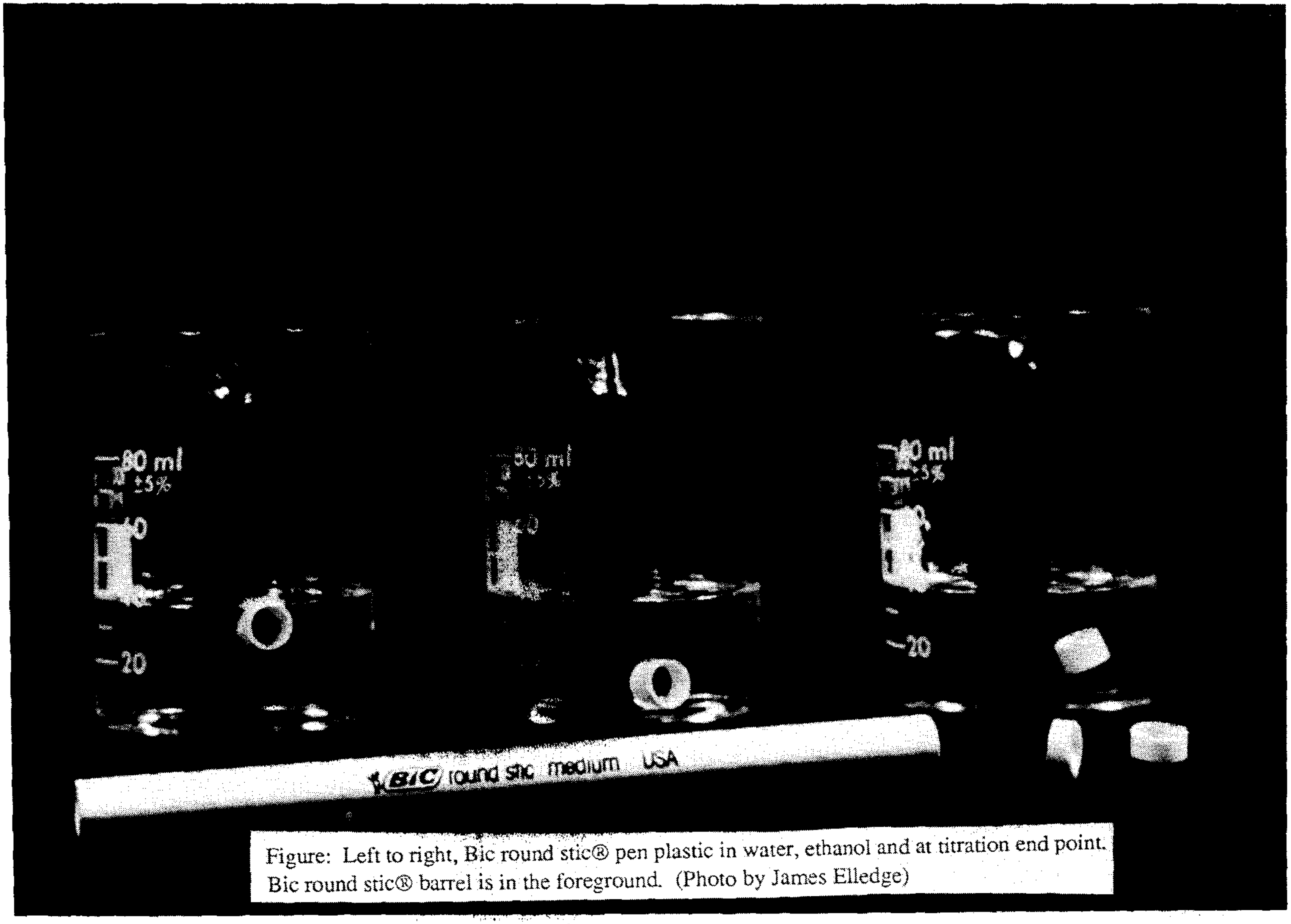

Figure: Left to right, Bic round stic® pen plastic in water, ethanol and at titration end point. Bic round stic® barrel is in the foreground. (Photo by James Elledge)

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